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# Pottery vessels and technology of "colouring materials" in the central-western Mediterranean (Sardinia, Italy) during the Middle Neolithic: an interdisciplinary approach combining use-wear and chemical-physical analysis

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## Abstract

Despite the wide occurrence of colour deposits adhering to the surfaces of several artefacts (specifically, pottery and lithic tools) in Early and Middle Neolithic sites of the Western Mediterranean regions, the *chaîne opératoire* of colouring materials has not been frequently addressed by systematic techno-functional studies. Particularly, the relationship between pottery function and coloured contents is generally overlooked.

In this paper, the use of colouring materials by Middle Neolithic (4500-4000 cal BC) societies in Sardinia (Italy) is investigated, focusing on the archaeological findings from the open-air settlement of Su Mulinu Mannu-Terralba (OR). The aims are to identify the kind of materials, to provide a first assessment of the production methods and to evaluate the role assigned to pottery vessels in processing, handling, and using colouring materials. For these purposes, we apply an interdisciplinary approach, combining analysis of lithic artefacts, use-wear analysis of pottery, archaeometric identification of the chemical and mineralogical composition of geomaterials and colour deposits on artefacts by PXRD, ATR-FTIR and SEM-EDS analyses, and biomolecular analysis of organic residues from pottery by GC-FID and GC-MS.

This study reveals the use of haematite-rich ochre as the exclusive red colour-producing geomaterial, processed *in situ* with basalt macro-tools. Use-wear associated with ochre deposits on pottery vessels points to the selection of some bowls and jars, respectively for processing and storing ochre as the single content or, possibly, as an ingredient of composite products. However, based on our data, the addition of organic materials to ochre is not definitely demonstrated. Beside the preparation of pigments, the occurrence of ochre as content in pottery vessels could be related to a broader range of purposes, encompassing both the technical and the symbolic realm. Overall, these results provide insights both on the technology of "colouring materials" and the use of pottery in the practices of Middle Neolithic groups.

## Keywords

Neolithic, Central-Western Mediterranean, Colouring materials, Ochre, Pottery use, Use-wear

## Highlights

- Investigation into the *chaîne opératoire* of ochre in a Middle Neolithic settlement
- Colour deposits and absorbed residues analysed by PXRD, ATR-FTIR, SEM-EDS and GC
- Use of haematite-rich ochre as the exclusive red colour-producing geomaterial
- Ochre processing with basalt stone tools, handling, storing and use in pottery
- Possible uses of ochre for multiple purposes in technical and symbolical practices

## 1. INTRODUCTION

The interest in colouring materials was deeply embedded in the technical and symbolic systems of Early and Middle Neolithic communities across central and northern Europe and the western Mediterranean regions (Tanda 2003; Bernabeu Aubán et al. 2008; Volante 2015; Hamon et al. 2016; Pradeau et al. 2016; Angeli et al. 2019; Gliozzo 2021). Considerable archaeological evidence exists for the peninsular and insular regions of present Italy, where the main recognised uses of colouring materials were as pigments: (i) in the decoration of artefacts such as pottery (Colombo, Boschian 2009; Fabbri et al. 2013; Giustetto et al. 2013; Quarta et al. 2018; Angeli et al. 2018, 2019; Armetta et al. 2023), anthropomorphic and zoomorphic figurines (Dal Rì et al. 2001; Fugazzola Delpino, Tinè 2002-2003; Colombo 2012; Ferrari, Pessina 2012; Grifoni Cremonesi, Pedrotti 2012; Gorgoglione et al. 2012; Bernabò Brea, Mazzieri 2014), personal ornaments (Dal Rì et al. 2001; Mazzieri, Micheli 2014), bone tools (Grifoni 1967; Colombo 2006), pebbles (Grifoni 1967; Cassano et al. 2003), (ii) in rock painting (Graziosi 1973, 1980), and (iii) possibly in decorating body and/or perishable objects, as suggested by colour traces on clay stamps (Serradimigni 2012; De Pascale 2014). Red pigments, such as ochre and cinnabar, were also a component of funerary behaviour, distinguishing several burials (Cipolloni-Sampò 1982; Santoni 1982; Dal Rì et al. 2001; Grifoni Cremonesi, Radmilli 2001; Odetti 2003; Ucelli Gnesutta 2003; Quarta et al. 2006; Zemour et al. 2017; Zemour 2019; Sparacello et al. 2019). Parallelly, the use of ochre for its multiple complementary properties, other than colouring (abrasive, drying, antimicrobial, preservative), is probably underestimated (Audouin, Plisson 1982; Pradeau et al. 2014: p. 653).

Pigments can be obtained from different geomaterials through various processes, including rubbing, reduction, grinding, sieving and sometimes decantation; they can be used as powders or in a liquid medium, sometimes with the addition of organic binders and/or mineral charges (Hodgskiss 2010; Pradeau et al. 2016; Horn 2018; Mastrotheodoros, Beltsios 2022; Domingo, Chieli 2021; Salomon et al. 2021). An in-depth archaeometric study revealed the exploitation of a wide range of colour-producing geomaterials during the 6th and the 5th millennium cal BCE in the north-western Mediterranean (Pradeau et al. 2016). Some clues of the technical actions carried out to obtain colouring materials are: raw and semi-processed geomaterials as chunks in the archaeological layers or powders in pottery vessels, lithic macro-tools bearing colour traces, and residues on the surfaces of pottery sherds and vases (for example, Grifoni 1967; Passeri 1970; Trump 1983; Camps 1988; Guerri 1988a-b; Barra et al. 1990; Germanà et al. 1990; Zamagni 2007; Fugazzola Delpino 2002; Tanda 2003; Ucelli Gnesutta 2003; Antona 2013; Pradeau et al. 2016; Daura et al. 2019). Nevertheless, the *chaîne opératoire* of colouring materials has not been frequently addressed by systematic techno-functional studies in European Neolithic contexts (García Borja et al. 2006; Domingo et al. 2012; Hamon et al. 2016; Pradeau et al. 2016; Defrasne et al. 2019). In the last fifteen years, an increasing attention has been deserved mainly to the archaeometric characterisation of pigments used in painted decorations or incrusted fillings in incised/impressed decorations of early and middle Neolithic pottery from peninsular Italy. These studies revealed the use of carbon black and manganese oxides as black pigments, haematite and sometimes cinnabar for red colours, and calcite, talc and hydroxyapatite for the white ones (Angeli et al. 2006, 2011; Giustetto et al. 2013; Quarta et al. 2018; Angeli et al. 2018, 2019). Contrariwise, colouring materials as residues of the original content of pottery vessels have been rarely investigated through archaeometric and functional approaches (Maniatis, Tsirtsoni 2004; Mioč et al. 2004; Gajić-Kvaščev et al. 2012; Pradeau 2015; Drieu et al. 2020). Consequently, despite the reported occurrence of colour residues on the surfaces of vessels, the relation linking pottery function and *chaîne opératoire* of colouring materials is generally overlooked**.**

In this work, we focused on the exploitation of colouring materials during Early and Middle Neolithic (6th-5th millennia cal BCE) in Sardinia (Italy), an insular region, whose geographical position far away from mainland and in the middle of the Western Mediterranean is particularly stimulating for the study of overseas human mobility and the subsequent diffusion of techniques and symbolic behaviour during the different phases of Neolithic (fig. 1; Lugliè 2018a; Fanti et al. 2018; Paba et al. 2021). After reviewing the available information, we investigated all the set of data traceable back to the cycle of production, storing and use of colouring materials from the middle Neolithic open-air site of Su Mulinu Mannu-Terralba (OR). The specific aims were: (i) to identify which colour-producing geomaterials were selected and used, (ii) to assess how colouring materials were processed, handled and utilised, (iii) to evaluate the role assigned to pottery vessels in the different phases of processing, storing and using colouring materials, in the broader framework of the functional structure of the ceramic assemblage. For these purposes, we applied an interdisciplinary functional approach, integrating use-wear analysis of pottery, analysis of lithic artefacts, archaeometric identification of the chemical and mineralogical composition of geomaterials and colour deposits on tools through powder X-ray diffraction (PXRD), attenuated total reflectance (ATR-FTIR) analyses, scanning electron microscopy – energy dispersive X-ray spectroscopy (SEM-EDS), and biomolecular analysis of organic residues from pottery by gas chromatography coupled with a flame ionization detector (GC-FID) and a mass spectrometer (GC-MS). A broader objective is to contribute with our results to the knowledge of the technology and potential multiple uses of colour-producing geomaterials in the domestic and ritual activities of Early and Middle Neolithic groups of Western Mediterranean regions.

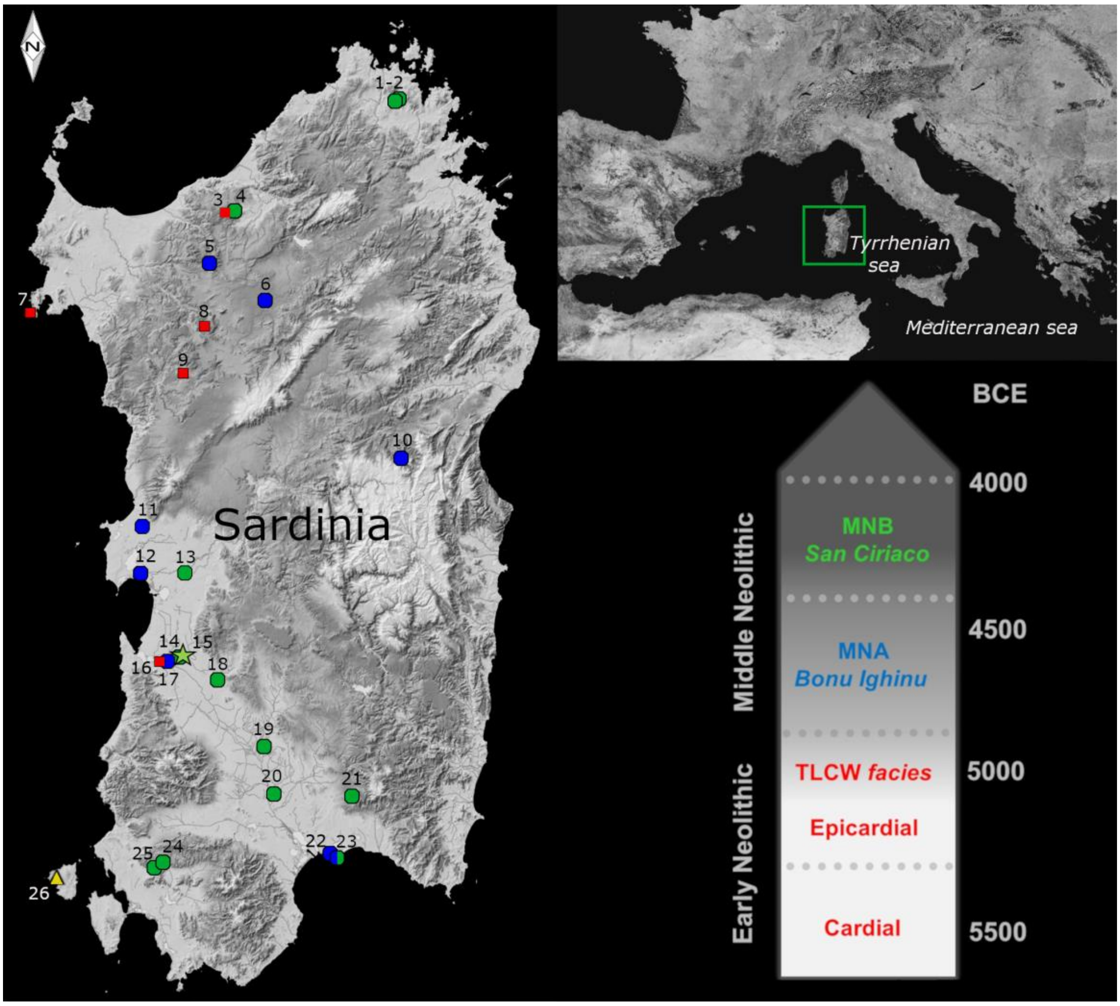


Fig. 1. Sites with reported use of colouring materials in Sardinia during the 6th and 5th millennium cal BCE (left) and chronology and phases of Sardinian Early and Middle Neolithic (right). Red squares: Early Neolithic sites; blue dots: Middle Neolithic A-Bonu Ighinu sites; green dots: Middle Neolithic B-San Ciriaco sites; green star: Su Mulinu Mannu-Terralba (OR); yellow triangle: geological source; 1-2: Li Muri and La Macciunitta proto-megalithic graves-Arzachena (SS), 3: Su Coloru cave-Laerru (SS); 4: Contraguda open-air site-Perfugas (SS); 5: Grotta dell'Inferno (cave)-Muros (SS); 6: Luzzanas or Sant'Antioco di Bisarcio (?)-Ozieri (SS); 7: Grotta Verde (cave)-Alghero (SS), 8: Sa Korona di Monte Majore cave-Thiesi (SS), 9: Filiestru cave-Mara (SS), 10: Grotta Rifugio (cave)-Oliena (NU), 11: Su Anzu (funerary site?)-Narbolia (OR); 12: Cuccuru is Arrius necropolis-Cabras (OR); 13: Su Cungiau de is Fundamentas open-air site-Simaxis (OR); 14: San Ciriaco open-air site-Terralba (OR), 15: Su Mulinu Mannu open-air site-Terralba (OR), 16: Bau Angius (burial?)-Terralba (OR); 17: Coddu is Abionis open-air site-Terralba (OR); 18: Puisteris open-air site-Mogoro (OR); 19: Sa Mandara open-air site-Samassi (CA); 20: Tanca Fara/Forada Campana open-air site-Villasor (CA); 21: Bingia Eccia (burial)-Dolianova (CA); 22: Grotta del Bagno Penale (cave)-Cagliari (CA); 23: San Bartolomeo cave-Cagliari (CA); 24: Grotta dei Fiori (cave)-Carbonia (CI); 25: Cannas di Sotto tomb-Carbonia (CI); 26: Becco geological source of iron oxides and manganese oxides-San Pietro island (CI). TLCW facies: Tyrrhenian linear carved ware *facies*; MNA: Middle Neolithic A; MNB: Middle Neolithic B (Map and image: L. Fanti).

## 2. The use of "colouring materials" in Sardinia between the 6th and the 5th millennium cal BCE: state of art and context of the study

The neolithisation of the island of Sardinia was promoted by the repeated movements of human groups across the western Mediterranean and particularly in the northern Tyrrhenian area, connecting mainland and insular regions first within the framework of the Cardial ware *koiné* and the subsequent Epicardial and Tyrrhenian linear carved ware *facies* (Early Neolithic, 6th millennium cal BC), then contributing to shape the features of the regional cultures through unceasing contacts and exchange during the middle Neolithic (5th millennium cal BCE) (fig. 1; Lugliè 2018a; Fanti et al. 2018, fig. 1,b; Paba et al. 2021; Lugliè 2020a).

As regards the use of colouring materials, the earliest evidence in Sardinia came from two Mesolithic sites, where red ochre was associated with burials (Lugliè 2018a); afterwards, it was integrated in technical activities and symbolic expressions of the human groups since the first Neolithic phases (table 1, supplementary file 1).

Table 1. Synoptic table about the use of colouring materials in Sardinia during the Early and Middle Neolithic phases. Unless otherwise specified, data refer to "red ochre". The numbers between parentheses correspond to the numbered sites in fig. 1 (see text and supplementary file 1 for detailed bibliographic references; *tentatively assigned to MNA phase).

| | Phase | | | |
|---|---|---|---|---|
| | **EN Cardial** | **EN Epicardial / Tyrrhenian linear carved ware phase** | **MNA *Bonu Ighinu*** | **MNB *San Ciriaco*** |
| | 6th millennium cal BCE | | 5th millennium cal BCE | |
| **Raw or semi-processed geomaterials** | Lumps in the sediment (8) | Semi-processed? (iron oxide, manganese oxide) (9) | | |
| **Processing tools** | | Basalt ground stone and handstone (9) | Basalt discoidal flat-handstone (10)* | Handstones (1?, 14) |
| **Storing in pottery vessels** | | Closed deep jars with handles (iron oxide, manganese oxide) (9) | | |
| **Use in pottery decoration and surface treatment** | Incrustation on cardial decoration (3, 8)<br>Application of red slip on surfaces (9) | Application of red slip on surfaces (3, 7, 9)<br>Painted pottery (7, 8) | White filling in impressed and incised decoration? (5, 22) | Filling in incised decoration: red (23); white(?) (4, 12, 18, 20)<br>Red filling in excised decoration or layer on all the surface? (24) |
| **Decoration or residues (?) on stone artefacts** | Red decoration on engraved pebbles or colour residues on stone stamps (17) | | | Red traces (painting?) on incised decoration of stone vessel (13) |
| **Residues on anthropomorphic figur(in)es** | | | Red traces on stone (11, 16) and bone (6) figurines, perhaps from destroyed burials | Red and bluish-black traces of coloured decoration on sculpted stone figures (19)<br>Red traces on bone figurine (25) |
| **Use in caves** | (8) | (9) | (10*, 23) | (23, 24) |
| **Use in funerary and ritual contexts** | | | Hypogeal graves (12)<br>Cave burial? (23)<br>Undetermined burials? (11, 16) | Proto-megalitic graves (1, 2)<br>Undetermined hypogeal burial (21)<br>Hypogeal burial (25)<br>Undetermined context (19) |
| **Use in open-air settlements** | Red decoration on engraved pebbles or colour residues on stone stamps (17) | | | Pottery decoration? (white residues?) (4, 12, 18, 20)<br>Unknown activities (red residues on tools: 14) |

| | |
|---|---|
| **Identification of geomaterials by archaeometric analysis** | Iron oxide<br>Manganese oxide<br>(9) |

Overall, the available data about the use of colouring materials during the Early and Middle Neolithic in Sardinia are discontinued and fragmentary, also due to the scantiness of contextualised findings from excavated sites. The identification of geomaterials is limited to a single Early Neolithic site (Tanda 2003) and the provenance of raw materials remains to be determined. A putative source for central-southern Sardinia has been identified at Becco-San Pietro Island (fig. 1), where geological deposits of haematite, limonite and manganese oxides occur in association with jasper outcrops, exploited as lithic raw material in several Neolithic sites (Lugliè 2005, 2020b; Lugliè et al. 2006, 2007). In the Early Neolithic, the use of colouring materials, mainly red ochre, is attested by dispersed residual chunks in the archaeological layers and storage of minerals in vessels; their main application was in decorating, which encompasses both technology and symbolic behaviour (Tanda 1980; Foschi 1982; Trump 1983; Lo Schiavo 1985, 1987; Foschi Nieddu 1987, 2002; Tanda 2003; Pitzalis et al. 2004; Lugliè, Pinna 2012; Fenu 2013; Fanti 2020). In the Middle Neolithic, the role of red colouring materials was deeply interconnected with the funerary and/or ritual sphere, whereas its use in domestic contexts and in artefact decoration seems to have been rare and dedicated to special items, such as anthropomorphic figure(in)es (Puglisi 1942; Atzeni 1975; Santoni 1982; Ferrarese Ceruti 1992; Guilaine 1996; Lilliu 1999; Antona 1998; Santoni 2000; Antona 2003; Lugliè 2004; Santoni 2012; Antona 2013; Lugliè 2017, 2018b; Antona 2020; Salis 2020). Lithic macro-tools were constantly employed for processing in almost all phases (Trump 1983; Agosti et al. 1980; Santoni et al. 1997). Some authors reported residues of white fillings in impressed or incised decorations of MNA and MNB pottery (Santoni et al. 1997; Lilliu 1999; Marras 1999; Fenu 2017). However, many Sardinian archaeological sites are frequently affected by post-depositional carbonate concretions (Fanti et al. 2018), whose appearance inside decorations could mimic intentional deposits. Therefore, these data should be considered with caution and need an accurate assessment to verify if the white residues resulted from a deliberate human activity or taphonomic alteration (Fanti 2019).

In this highly incomplete framework, the site of Su Mulinu Mannu-Terralba (TMM), offered the opportunity to learn more about the production and use of colouring materials by the MNB groups. This open-air site, located near the eponym site of San Ciriaco-Terralba (OR), is one of the few excavated and radiocarbon dated MNB settlements (Fanti 2015), characterised by irregular pits, such as the structure S2, containing large amounts of pottery sherds, ground stone and chipped stone artefacts, faunal and botanical remains (fig. 2; Fanti et al. 2018: fig. 2, c). Previous studies of this context enhanced knowledge of the lifeways and household activities of Sardinian MNB societies (Ucchesu et al. 2017; Fanti et al. 2018; Fanti 2019). The whole functional structure of the pottery assemblage was determined through interdisciplinary research, allowing the direct identification of the resources processed, stored, and consumed in the site through the biomolecular and isotopic analysis of organic residues absorbed into the vessel walls: mainly, adipose ruminant fats, dairy and plant products (Fanti 2015; Fanti et al. 2018). Additionally, in some vessels, direct evidence of the early content was the presence of red or yellow-orange deposits adhering to the exterior and/or interior surfaces, suggesting the use of pottery in relation with colouring materials (Fanti 2015). Recently, similar red deposits have been detected also on other categories of remains from the same site, such as lithic macrotools, together with chunks of colour-producing geomaterials. Thus, in the site of TMM, several clues on both the production and use of colouring materials were associated in a MNB household context, allowing the application of an interdisciplinary functional approach to start addressing this issue in a more systematic way.


Fig. 2. Su Mulinu Mannu-Terralba (OR): the S2 pit under excavation. On the left, in section, the fragment of the grinding slab TMM12952 (TMM_gs1), bearing red residues (Photo: C. Lugliè).

## 3. Materials and methods

### *3.1 Analysis of lithic artefacts and geological samples*

The macrolithic collection of TMM, including several grinding tools, such as grinding slabs, flat-handstones and pestles, has been examined in its entirety (30 elements). After a first assessment of the lithic assemblage, the items showing coloured residues (red and/or white) were selected for a more detailed analysis. Although the deposits were often easily recognisable with the naked eye, all these elements were submitted to observation through a stereomicroscope (Euromex ZE 1671, 10x to 60x), in order to detect even very marginal traces. As a result, a total of nine fragments of different grinding slabs and a flat handstone with red deposits, and one pestle with red and white residues, all made on volcanic rocks (mainly basalt), were identified (fig. 3).
Three small chunks of red mineral (≤ 1 cm) were found in association with other archaeological remains (fig. 3). These cohesive materials with good pigmenting properties (Defrasne et al. 2019) had mostly soft hardness (Mohs scale 1 and 2) and their surface colour was similar to the colour of the red powder they produced.
A sample of this geomaterial (TMM_geo_cm), together with colour residues on two grinding slabs (TMM_gs1_rd, TMM_gs2_rd) were analysed by XRD, ATR-FTIR, and SEM-EDX, while the white residue on the pestle (TMM_pst_wd) was analysed by SEM-EDX due to its low amount (table 2). Furthermore, a geological basalt cobble (TMM_geo_bas), unearthed at the same site, was

analysed as a reference sample for the mineralogical composition (XRD, ATR-FTIR) of the ground stone tools together with a sample of the archaeological soil (TMM_soil, from the US1029), in order to discriminate potential contaminations in the red deposits on the artefacts, arising from the contact with the soil and/or the attrition on the lithic tools in the mechanical processing of colouring materials.

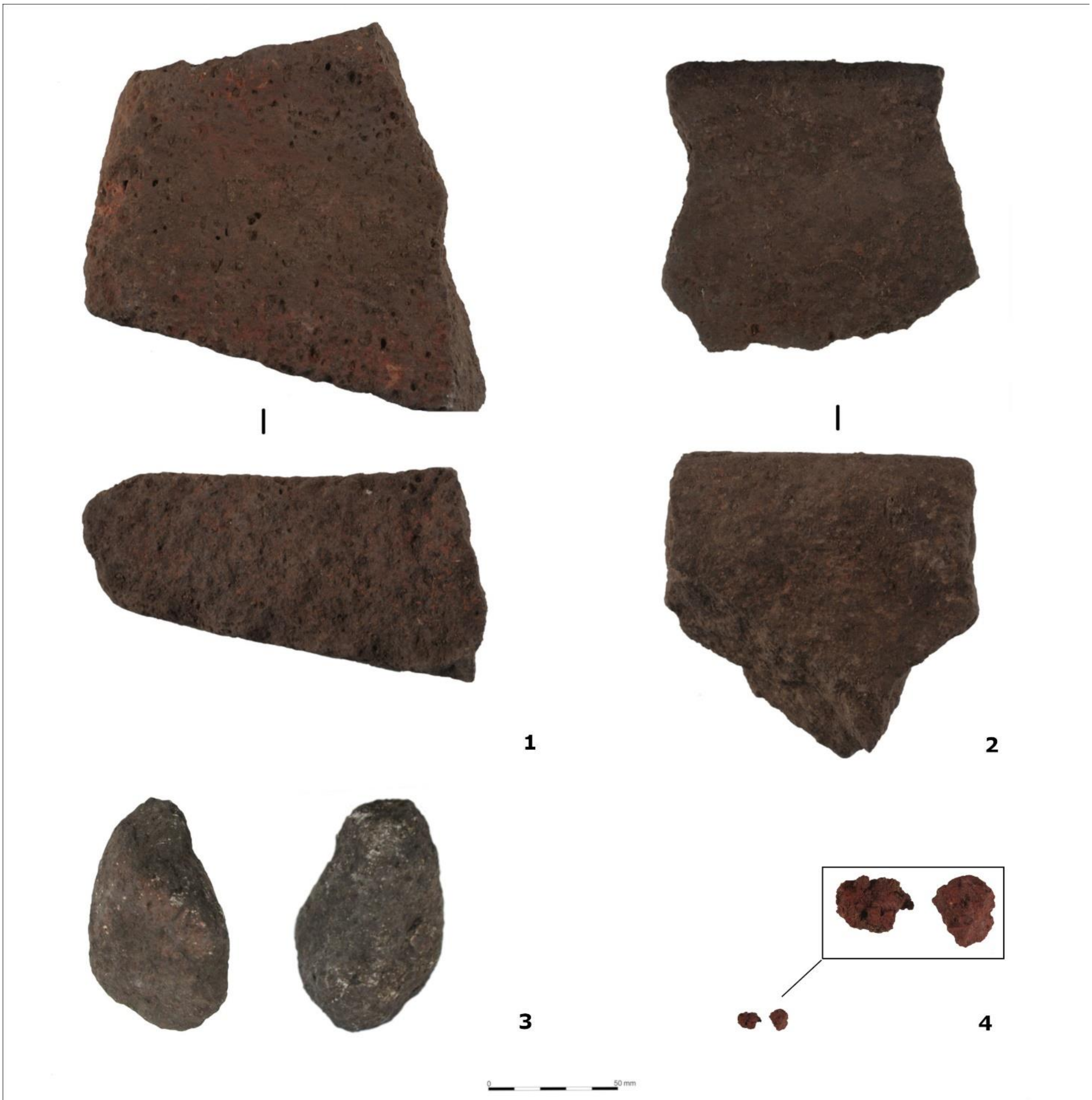


Fig. 3. Lithic macro-tools and geomaterials from the TMM site: 1) TMM12952 (TMM_gs1) and 2) TMM F32971 (TMM_gs2): grinding slab fragments, workface (showing colour residues) and section; 3) TMM F36516 (TMM_pst): pestle; 4) TMM_geo_cm: small chunks of red mineral (Photos and image: B. Melosu).

### *3.2 Functional analysis of the pottery assemblage*

Pottery from TMM (more than 5000 sherds) was highly fragmented, but systematic refitting led to reconstruct a minimum number of 138 vessels with different morphology and dimensions: bowls, jars, ladle-bowls, cooking pots and big-size vessels (Fanti et al. 2018). Functional analysis was conducted following the methodology fully detailed in Fanti et al. 2018, Fanti 2019, combining morpho-typology, morphometry and use-wear data. In this work, use-wear was investigated through macroscopic and microscopic observation by stereomicroscope (low magnification: 10x-60x), with the specific aim of recording attritions and colour deposits. As observed in the literature, the occurrence of red deposits on archaeological artefacts can result from intentional application, indirect staining, or natural deposition caused by taphonomic alteration (Howard 2010; Velliky et al. 2018). In order to distinguish among these three potential processes of formation of red deposits on the TMM vessels, the same principles applied for the discrimination of use-alteration from post-depositional unintentional traces were taken into consideration: (i) presence of a discrete pattern on the surfaces (evaluation of position and distribution of the residues), (ii) continuity of traces on adjoining sherds, (iii) site-specific taphonomic alterations (Skibo 2013; Vieugué 2014; Fanti 2019; Debels et al. 2023). Specifically, the position (interior/exterior surface, upper/medium/lower portion of the vessel), appearance (colour, morphology, thickness) and distribution (localised, covering) of residues, and attritions (linear scratches, abraded areas) on the pottery surfaces were recorded and considered in relation with vessel morphology and morphometry and its main technological features (type of paste, surface treatment).

Colour residues (TMM_pvX_xx_rd) and pottery (TMM_pvX_xx_p) were sampled, when possible, respectively for archaeometric characterisation and organic residue analysis, as detailed in the following paragraphs.

### *3.3 Archaeometric characterisation of colour residues*

The archaeometric characterisation of the colour residues on the lithic tools and pottery vessels involves the study of the crystalline phases, the elemental composition, the micromorphology and the analysis of eventual residues of organic binders (Mastrotheodoros, Beltsios 2022).

Several analytical techniques are currently used for the determination of the mineralogical and elemental composition of colouring materials (Dayet 2021; Domingo, Chieli 2021; Mastrotheodoros, Beltsios 2022; Popelka-Filcoff, Zipkin 2022). The sampling and the choice of techniques for the analysis of different materials from TMM was influenced by the effective amount of red deposits on the artefact surfaces: in some cases, only one technique was applicable (e.g., SEM-EDS analysis), due to the small amount of residues (table 2). The sampling was conducted directly on the colour residues or by separately removing different layers from an area of the pottery vessel where a colour residue was identified (fig. 4, table 2). In the sampling of pottery for the analysis of absorbed residues, the exposed surfaces are usually discarded to minimise contamination from soil and handling of sherds (Heron et al. 1991; Whelton et al. 2021). This approach was carried out to reach the inner part of the pottery sample for organic residue analysis, but the outer layers were used in order to get information on the inorganic components (fig. 4; see 3.4 for further details).

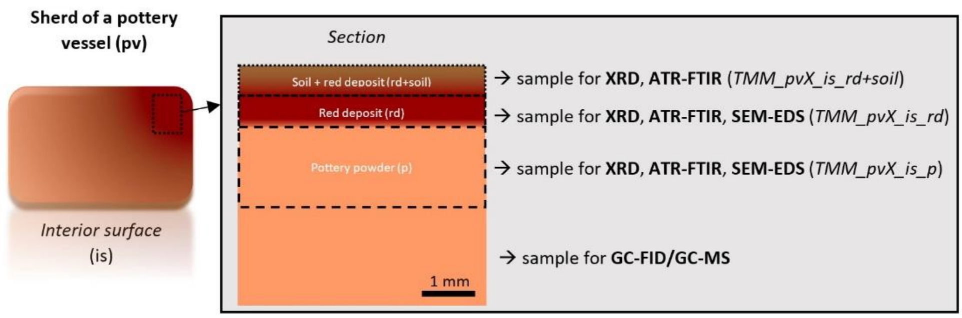


Fig. 4. Sketch of the sampling approach used on the pottery sherds to select the samples for the archaeometric analyses (Image: V. Mameli, L. Fanti).

XRD analysis was performed to identify the mineralogical composition of the chunk of red colouring geomaterial (TMM_geo_cm), the colour residues on lithic tools (TMM_gsX_rd) or pottery vessels (TMM_pvX_xx_rd), the pottery matrix (TMM_pvX_xx_p), and some reference materials (TMM_soil, TMM_geo_bas).
ATR spectra were acquired to confirm the mineralogical composition through the identification of the main vibrational modes associated with the functional groups.
SEM-EDS analysis was applied in order to observe the morphology of red deposits and to identify the chemical composition of the colour materials, investigating, as an example, the occurrence of iron (pointing to an iron oxydes/hydroxydes-based pigment) and/or mercury (indicating the presence of cinnabar), possibly in association with other significant elements (calcium, manganese, etc.; Domingo et al. 2012; Pradeau et al. 2016; Gliozzo 2021).
Detailed information on the instruments and protocols is provided in Supplementary file 2.

### *3.4 Biomolecular analysis of organic residues from pottery vessels*

Many attempts to detect residues of organic binders, eventually added to the pigments, have been reported in the literature, especially on prehistoric rock paintings; nevertheless, their identification remains highly challenging (Spades, Russ 2005; Domingo, Chieli 2021). This can be a crucial issue also for the residues adhering to pottery vessels, especially if they are preserved in very low amount and/or as a thin layer, because they would be difficulty separated from the adhering sediment, often containing organic compounds (Heron et al. 1991; Whelton et al. 2021). Unfortunately, the amount of red deposits from the TMM artefacts was insufficient for extraction and GC analysis. However, an advantage considering residues from vessels is the capability of pottery to absorb liquid or viscous contents, whose traces could have been preserved into the pottery walls.
Organic residues can be absorbed and often altered during all the vessel lifecycle, depending on multiple factors, such as the physical properties of pottery, the modalities of use, the kind of contents, the environmental and burial conditions, and the post-excavation practices (Evershed 2008a-b: Correo-Ascencio, Evershed 2014; Roffet-Salque et al. 2017; Hammann, Cramp 2018; Fanti et al. 2018). The presence of colour residues on the pottery surfaces provides direct evidence of the (last) vessel use; nonetheless, this could not correspond to the main function(s) (Skibo 2013; Roffet-Salque et al. 2017). The biomolecular signal from vessels with colour residues could derive from an organic medium added during the preparation of colouring materials (Drieu et al. 2020), and/or from other content(s), absorbed during the last steps of vessel production or different vessel use(s) (Evershed 2008b; Drieu et al. 2019; Reber et al. 2019; Miller et al. 2020). By exploring the mineral and organic residues in connection with use-wear and morphometric features, we can better assess an eventual vessel multifunctionality or, conversely, a specific selection of pottery for the exclusive purpose of handling and storing colouring materials.

After carefully sampling the sediment/residue interface, the colour residues and the interior surface with different clean scalpel blades (fig. 4), the underlying pottery was mechanically sampled and submitted to preparation and GC-FID/GC-MS analysis following established protocols (Supplementary file 2; Correo-Ascencio, Evershed 2014; Papakosta et al. 2015; Drieu et al. 2020; Reber 2021; Suryanarayan et al. 2022).

Table 2. – List and description of the samples and related analyses (1: ATR-FTIR carried out with Agilent Cary 630; 2: ATR-FTIR carried out with Bruker Vertex 70; see Supplementary file 2 for detailed information).

| n. | Pottery vessel /Lithic tool Id. | Sample Id. | Sample description | Analytical techniques | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | *XRD* | *ATR-FTIR (1)* | *ATR-FTIR (2)* | *SEM-EDS* | *GC-FID/ GC-MS* |
| 1 | | TMM_soil | Soil sample from US1029 | X | X | | X | X |
| 2 | | TMM_geo_cm | Chunk of red colouring geomaterial (geo_cm) | X | X | | X | |
| 3 | | TMM_geo_bas | Geological basalt (geo_bas) cobble | X | X | | | |
| 4 | TMM12952 | TMM_gs1_rd | Red deposit (rd) on basalt grinding slab (gs) | X | X | X | X | |
| 5 | TMM F32971 | TMM_ gs2_rd | Red deposit (rd) on basalt grinding slab (gs) | X | X | X | X | |
| 6 | TMM F36516 | TMM_pst_wd | White deposit (wd) on basalt pestle (pst) | | | | X | |
| 7 | TMM1213 | TMM_pv1_is_rd | Red deposit (rd) on the interior surface (is) of pottery vessel (pv) | | | X | X | |
| | TMM1213 | TMM_pv1_es_rd | Red deposit (rd) on the exterior surface (es) of pottery vessel (pv) | | | X | X | |
| 8 | TMM1877<br>TMM F9286 | TMM_pv2_is_rd | Red deposit (rd) on the interior surface (is) of pottery vessel (pv) | X (1877) | X (1877) | X (F9286) | X (F9286) | |
| | TMM1877 | TMM_pv2_is_p | Pottery powder (p) from the vessel interior surface (is) | X | X | | | X |
| 9 | TMM12948 | TMM_pv3_is_rd+soil | Red deposit with soil residue (rd+soil) on the interior surface (is) of pottery vessel (pv) | X | X | | | |
| | TMM12948 | TMM_pv3_is_rd | Red deposit (rd) on the interior surface (is) of pottery vessel (pv) | X | X | | X | |
| | TMM12948 | TMM_pv3_is_p | Pottery powder (p) from the vessel interior surface (is) | X | X | | | X |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 10 | TMM12622 | TMM_pv4_is_yd | Yellow deposit (yd) on the interior surface (is) of pottery vessel (pv) | X | X | X | X | |
| | TMM12622 | TMM_pv4_is_p | Pottery powder (p) from the vessel interior surface (is) | X | X | | | X |
| 11 | TMM12362 | TMM_pv5_is_p | Pottery powder (p) from the vessel interior surface (is) | | | | | X |
| 12 | TMM12645 | TMM_pv6_is_p | Pottery powder (p) from the vessel interior surface (is) | | | | | X |
| 13 | TMM2398 | TMM_pv7_is_p | Pottery powder (p) from the vessel interior surface (is) | | | | | X |
| *total number of analysed samples for each technique* | | | | 12 | 12 | 6 | 10 | 7 |

*ATR-FTIR(1): Agilent Cary 630*
*ATR-FTIR(2): Bruker Vertex 70 FTIR*

## 4. RESULTS AND DISCUSSION

### *4.1 Elemental, structural, and mineralogical identification*

The presence of red deposits on different basalt ground tools (grinding slabs, flat-handstone and pestle: fig. 3) demonstrates *in situ* processing of the colouring minerals (Domingo et al. 2012). The three small mineral chunks probably result from the reduction of geological materials during their processing into red colouring powders, perhaps with a back-and-forth motion (Shoemaker et al. 2017). Unfortunately, more specific traces, clearly referable to the technique of reduction and processing (Hodgskiss 2010), have not been identified on their surfaces.

Twelve samples were analysed by PXRD, including three reference materials (TMM_soil, TMM_geo_cm, TMM_geo_bas), five red deposits from both lithic tools (TMM_gs1_rd, TMM_gs2_rd) and pottery vessels (TMM_pv2_is_rd, TMM_pv3_is_rd+soil, TMM_pv3_is_rd), one yellow deposit from a pottery vessels (TMM_pv4_is_yd), and the pottery powder as reference for the colour deposits (TMM_pv2_is_p, TMM_pv3_is_p, TMM_pv4_is_p). XRD analyses identified haematite in the five red deposits (TMM_gs1_rd, TMM_gs2_rd, TMM_pv2_is_rd, TMM_pv3_is_rd+soil, TMM_pv3_is_rd) adhering to the ground stones and the pottery surfaces, associated with quartz, micas, feldspars, and probably small quantities of pyroxenes (fig. 5, table 3). The mineralogical composition of the chunk of red ochre (TMM_geo_cm) is dominated by haematite, quartz and micas. On the contrary, haematite seems absent (or lower than the detection limit of the technique) from the soil (TMM_soil), the yellow deposit from TMM12622 (TMM_pv4_is_yd), all the interior pottery surfaces and the geological basalt sample used as reference for the mineralogical composition of the grinding slabs. The soil sample is composed by quartz, micas, and feldspar, while pyroxenes are found besides feldspars in the geological basalt rock. The small amounts of pyroxenes in the red deposits from the artefacts could have been incorporated in the powder during the processing of ochre, because of the attrition with the basalt macrotools. However, these traces are very marginal, highlighting the effectiveness of the basalt ground stones in the mechanical use for processing the ochre material. Parallelly, the ubiquitarian occurrence of feldspars in all the red deposits, regardless the artefact (ground stone or pottery), points to a contamination from the soil (fig. 5). Additional diffraction peaks probably ascribable to calcite were detected in the red deposit from a basalt ground stone (TMM_gs1_rd). The mineralogical composition of the yellow deposit from TMM12622 was made up by quartz, micas and feldspars, and the presence of other iron-based yellow pigments (*e.g.*, goethite) was not revealed.

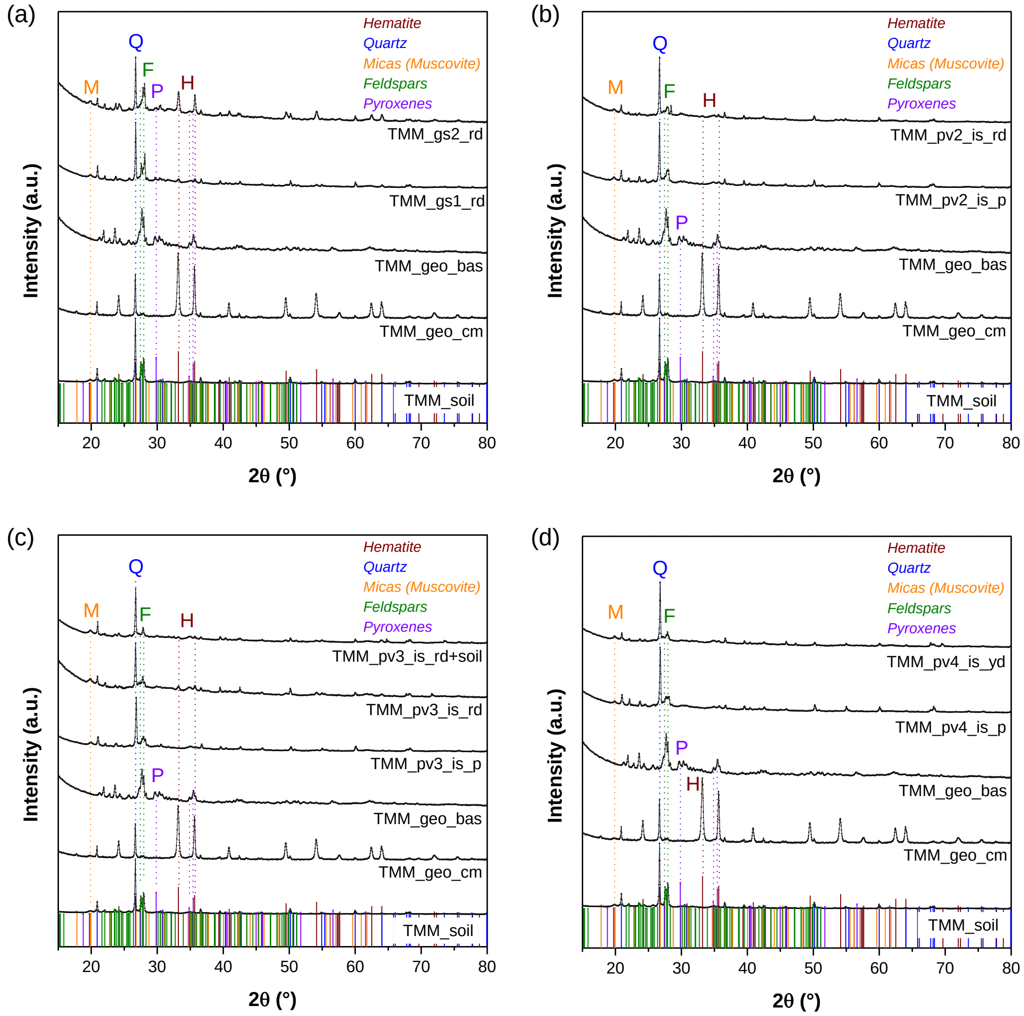


Fig. 5. Powder XRD patterns of the red and yellow deposits sampled on the surfaces of the basalt grinding slabs (TMM_gs1_rd (TMM12952), TMM_gs2_rd (TMM F32971) and pottery vessels (TMM_pv2_is_rd (TMM 1877), TMM_pv3_is_rd+soil and TMM_pv3_is_rd (TMM12948), TMM_pv4_is_yd (TMM12622), and of some reference materials, such as the soil sediment (TMM_soil), the chunk of red mineral (TMM_geo_cm), the geological basalt cobble (TMM_geo_bas), and the interior surface of the pottery (TMM_pv2_is_p, TMM_pv3_is_p, TMM_pv4_is_p). The coloured and dashed lines have been added to depict the most intense diffraction peaks of the different mineralogical phases.

Table 3. Results of XRD analysis (X: presence, -: absence; u.a.: possible presence of small quantities).

| Sample | Haematite | Quartz | Micas | Feldspars | Pyroxenes | Calcite |
|---|---|---|---|---|---|---|
| TMM_soil | - | X | X | X | - | - |
| TMM_geo_cm | X | X | X | - | - | - |
| TMM_geo_bas | - | - | - | X | X | - |
| TMM_gs1_rd | X | X | X | X | *u.a.* | *u.a.* |
| TMM_gs2_rd | X | X | X | X | *u.a.* | - |
| TMM_pv2_is_rd | X | X | X | X | *u.a.* | - |
| TMM_pv2_is_p | - | X | X | X | *u.a.* | *u.a.* |
| TMM_pv3_is_rd+soil | X | X | X | X | *u.a.* | - |
| TMM_pv3_is_rd | X | X | X | X | *u.a.* | - |
| TMM_pv3_is_p | - | X | X | X | - | - |
| TMM_pv4_is_yd | - | X | X | X | *u.a.* | - |
| TMM_pv4_is_p | - | X | X | X | X | - |

*u.a. = uncertain attribution due to the very weak diffraction peaks*

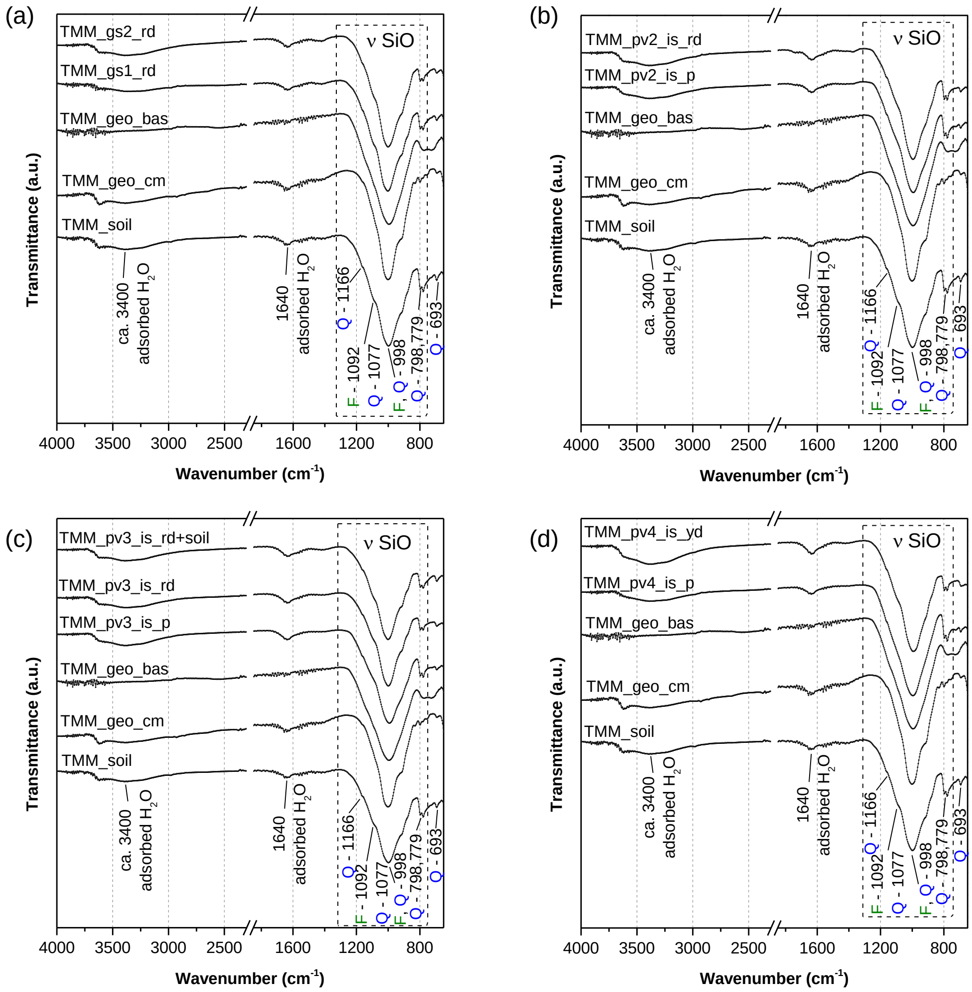


Fig. 6. ATR spectra of the red and yellow deposits from the basalt grinding slabs (TMM_gs1_rd (TMM12952), TMM_gs2_rd (TMM F32971), pottery vessels (TMM_pv2_is_rd (TMM1877), TMM_pv3_is_rd+soil and TMM_pv3_is_rd (TMM12948), TMM_pv4_is_yd (TMM12622)), and some reference materials, such as the soil sediment (TMM_soil), the chunk of red mineral (TMM_geo_cm), the geological basalt cobble (TMM_geo_bas), and the interior surface of the pottery (TMM_pv2_is_p, TMM_pv3_is_p, TMM_pv4_is_p).

The same twelve samples were analysed also by ATR and the spectra are shown in fig. 6. Typical vibrational modes of adsorbed water (ca. 3400 $cm^{-1}$, 1640 $cm^{-1}$ corresponding to the O-H stretching and H-O-H bending, respectively), quartz and silicates (between 1200-700 $cm^{-1}$) were identified, in agreement with the mineralogical composition obtained by PXRD. Although all samples are characterized by Si-O bonds, slight differences are visible in the spectra depending on the mineralogical composition. For instance, no quartz vibrational modes are visible in the geological basalt rock, which also exhibit a wider band at 1000 $cm^{-1}$, probably due to the superposition of feldspars and pyroxenes signals. On the contrary, the spectrum of the chunk is sharper compared to the others. Moreover, it is worthy to note that the spectra of the sediment, pottery surfaces and red deposits are quite similar, due to the presence of the same crystalline phases. No additional information was obtained for the yellow deposit (TMM_pv4_is_yd) found in the pottery TMM12622, that did not feature additional vibrational modes in comparison with the other samples. Therefore, the yellow deposit cannot be ascribed to the presence of organic compounds detectable by ATR or crystalline phases detectable by PXRD, or they are present in a concentration below the detection limit of the two analytical techniques.Some selected red deposits on both lithic tools and pottery vessels, three of them (TMM_gs1_rd, TMM_gs2_rd, TMM_pv2_is_rd) already analysed by XRD and ATR-FTIR(1), two additional samples (TMM_pv1_is_rd, TMM_pv1_es_rd), whose amount was too low for a complete characterization, and the yellow deposit (TMM_pv4_is_yd) were analysed by ATR with another instrument that permit to study the spectral region between 650 and 350 $cm^{-1}$, in which the typical vibrational modes of Fe-O bonds are found, in order to confirm the presence of haematite and further investigate the composition of the yellow deposit (fig. 7). The spectra of the deposits appear similar for all samples but that of the yellow deposit, in agreement with the XRD assignment of the same colouring material for the red deposits. In detail, vibrational modes at about 520 $cm^{-1}$, between 450 $cm^{-1}$ and 420 $cm^{-1}$, and 400 $cm^{-1}$, compatible with the haematite $E_u$ and $A_{2u}$ vibrations (Cornell et al. 1996), are visible in all red deposits. Furthermore, for comparison, commercial pure haematite samples (purchased from Sigma Aldrich) in form of both micro (Hem_micro) and nanoparticles (Hem_nano) were analysed. Slight differences in the position of these bands are observed with respect to those recorded for the red deposits, which might be due to differences in sizes and shapes of the hematite particles, and possible cation substitution, as widely reported in the literature (Cornell et al. 1996). Therefore, despite the limits deriving from the overlapping with the vibrational modes of the main mineralogical phases (quartz, silicates, aluminosilicates) in these red deposits, the ATR spectra suggest a compatibility with the presence of hematite.

The spectrum of the yellow deposit (TMM_pv4_is_yd) was also compared with those of two iron oxyhydroxides synthesised according to established procedures (Cornell et al. 1996), whose typical colours are yellowish, *i.e.*, schwertmannite (Schw, $Fe_{16}O_{16}(OH)_y(SO_4)_z \cdot nH_2O$) and goethite (Goet, FeOOH). Schwertmannite is a poorly-crystalline Fe(III) oxyhydroxy-sulfate phase, which precipitates from acidic sulfate-rich waters (Schoepfer, Burton 2021), while goethite more easily occurs in nature, being one of the most thermodynamically stable iron oxyhydroxides at ambient temperature and the final phase of many transformations (Cornell et al. 1996).

Unfortunately, it is not possible to conclude on the presence of such iron oxyhydroxides in the sample TMM_pv4_is_yd, due to the copresence of high amount of different crystalline phases, as evinced by XRD, which contribute to the complexity of the spectrum, and absence of information on the crystalline Fe-bearing phases. Indeed, the main bands present in the spectrum, i.e., those at about 800 $cm^{-1}$ and 470 $cm^{-1}$ and the shoulder in the range 600-550 $cm^{-1}$ are ascribable to the symmetric stretching of Si-O-Si groups of quartz and the silicate phases, and their bending modes, respectively.

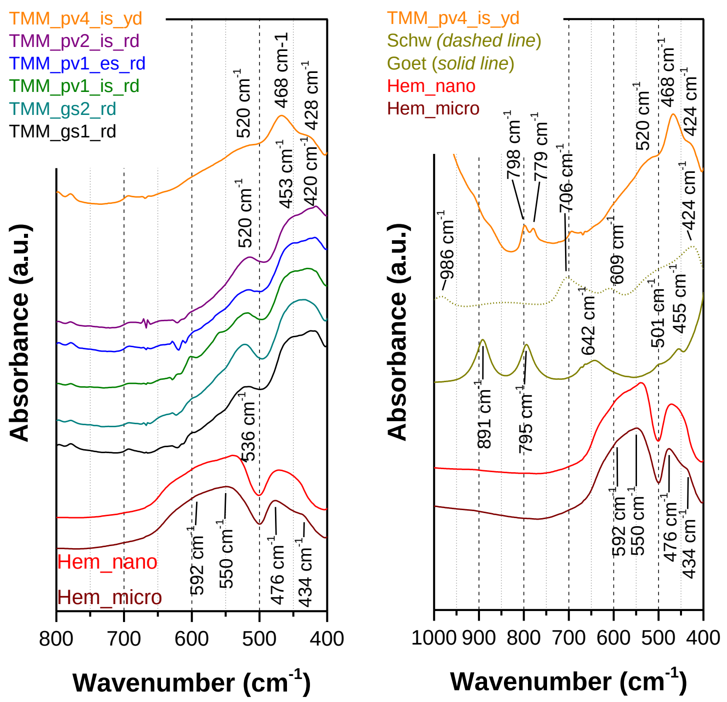


Fig. 7. ATR spectra in the region 700-350 cm$^{-1}$ of the red and yellow deposits from the basalt grinding slabs (TMM_gs1_rd (TMM 12952), TMM_gs2_rd (TMM F32971), pottery vessels (TMM_pv1_es_rd, TMM_pv1_is_rd (TMM 1213), TMM_pv2_is_rd (TMM 1877), TMM_pv4_is_yd (TMM 12622) and reference materials (Hem_nano, Hem_micro, Schw, Goet) (Image: V. Mameli).

Seven out of the twelve samples analysed by XRD and ATR-FTIR were characterised also by SEM-EDS to determine the elemental composition. In addition, two more samples of red deposits from a pottery vessel (the same analysed by ATR-FTIR(2), TMM_pv1_is_rd, TMM_pv1_es_rd (TMM1213) and the white deposit from the basalt pestle (TMM_pst_wd) were analysed. EDS analyses showed the presence of Fe in the mineral chunk (weight concentration > 60%) confirming the presence of a colour material based on α-$Fe_2O_3$ (haematite), together with Si and Al, in agreement with the XRD and ATR-FTIR analyses. This composition suggests the identification of this sample as ochre, specifically an iron oxide rich (> 80%wt. of $Fe_2O_3$) mixture with aluminosilicate phases (6-9%wt. of $SiO_2$, 3-5%wt. of $Al_2O_3$) (Siddall 2018; Mastrotheodoros, Beltsios 2022). Moreover, Fe was present in all the analysed red deposits from grinding slabs (TMM_gs1_rd, TMM_gs2_rd) and pottery (TMM_pv1_is_rd, TMM_pv1_es_rd, TMM_pv2_is_rd, TMM_pv3_is_rd) (fig. 8, table 3), with mean atomic concentration (in the range 3-10%) higher than that of the soil sample (TMM_soil, about 2%). Since no evidence for the presence of Hg was found, it is possible to hypothesise the absence of cinnabar as red pigment, besides haematite. Therefore, only one type of red mineral (haematite-rich) seems to have been used in the TMM site.

SEM-EDS analysis on the white deposit from the basalt pestle (TMM_pst_wd) clearly points out the presence of Ca and P as major elements (about 12% and 7% as mean atomic concentrations, respectively), that based on the semi-quantitative analysis (table 4) does not arise from a soil contamination (mean atomic concentrations lower than 2%), and can be ascribed to calcium phosphate, the main component of bones (Vieugué et al. 2015). A further confirmation of the presence of bone residues is associated with the detection of small amount of F and the typical fibrous structure (fig. 9).

The elemental composition of the yellowish sample TMM_pv4_is_yd reveals the presence of Fe with percentages analogue to those observed for the red deposits, but unfortunately this cannot be interpreted as a proof of the presence of Fe-bearing colouring materials, since no other evidence was found, either by XRD or ATR-FTIR.

In order to better visualise the differences among the samples, based on the elemental composition, the atomic ratios for the most informative elements, *i.e.*, Fe/(Si+Al), Ca/(Si+Al), and P/(Si+Al), were calculated as reported in table 5 and a representative graph of Ca/(Si+Al) *versus* Fe/(Si+Al) was obtained (fig. 10). As expected, the white deposit and the mineral chunk are opposite in the plot and separate with respect to the other samples; the red deposit collected from the external surface of the pottery vessel TMM1213 (TMM_pv1_es_rd) shows a higher relative Ca and P content than the other red deposits.

A higher percentage of Ca and P was also detected in the red deposit from one of the basalt grinding slab (TMM_gs2_rd), in agreement with bone processing, as suggested by the possible presence of calcite by XRD analysis, a typical component of bone tissues.

The absence of calcite in the mineral chunk indicates that this mineral was not a component of the geological sample. Some studies showed the practice of adding other minerals (clays and/or calcite) to ochre during different phases of the production sequence of the pigments (García Borja et al. 2004; Domingo et al. 2012). The detection of very low quantities of Ca by SEM-EDS in all the red deposits (except for weak traces only on a grinding slab or in the external surface of the pottery vessel TMM1213), suggests that no charge was deliberately mixed in manufacturing the red colouring materials at TMM. The distribution of powdered bone (white deposits) and red deposits on the pestle and the occurrence of weak traces of calcite in the red deposit from a grinding slab point to distinct and sequential uses for grinding different materials, not necessarily associated in the same final product. Therefore, these findings highlight the multifunctionality of the macrolithic tools in the TMM site.

The production of bone powder is attested in several ethnographic and archaeological contexts, for various purposes, ranging from consumption (as a component of meals or medicinal remedies) to technical application, *e.g.*, as white colouring material (Giustetto et al. 2013; Vieugué et al. 2015; Ge et al. 2021). However, comparable white deposits were not found on pottery vessels nor other artefacts at TMM. Although the occurrence of white fillings on MNB decorated vessels is reported in the literature (see 2 paragraph), at this stage of research, the eventual use of bone powder as a white colouring material in the TMM site cannot be further discussed.

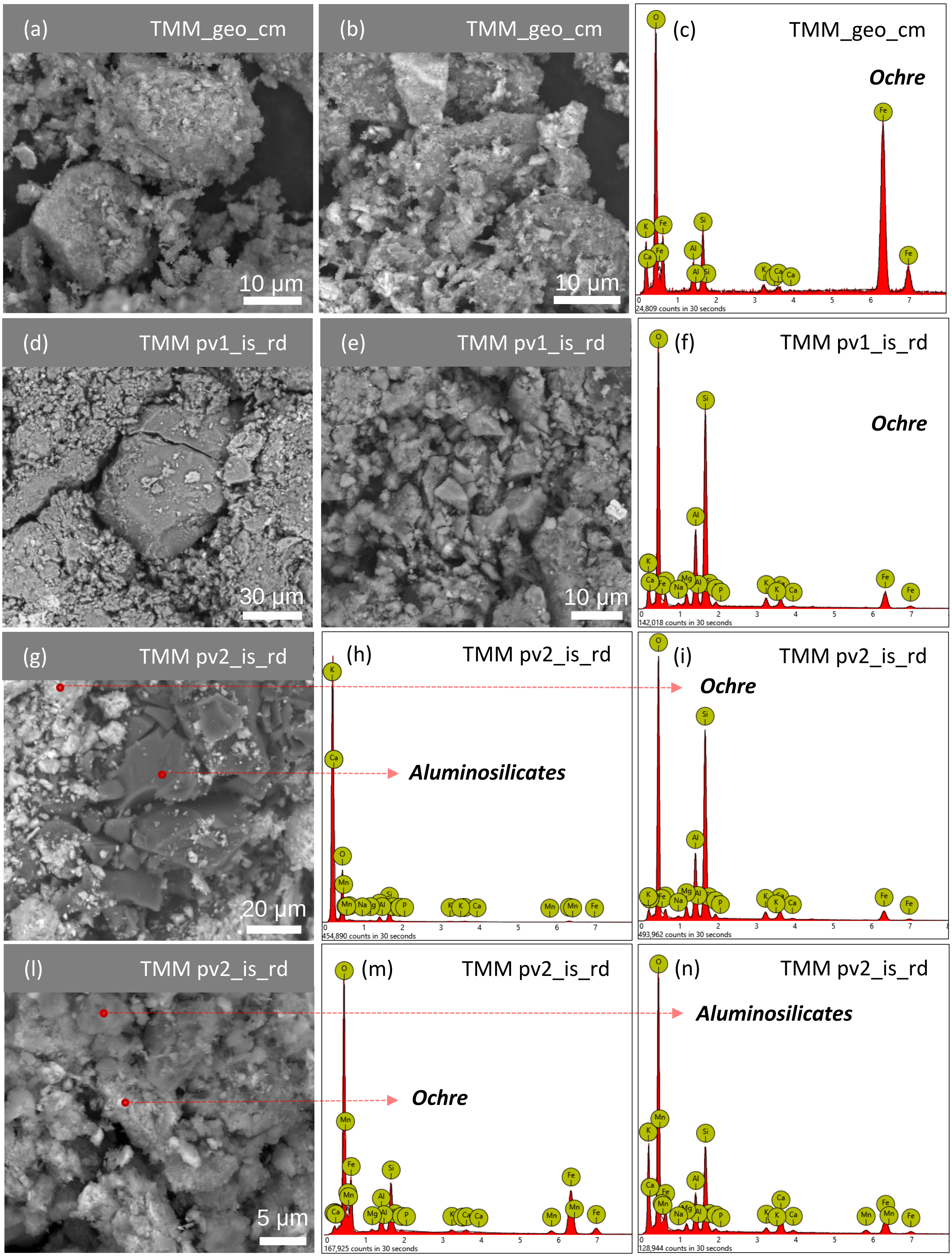


Fig. 8. Backscattered SEM micrographs (a, b, d, e, g, l) of some ochre residues and EDS spectra (c, f, h, i, m, n) (Image: B. Melosu, V. Mameli).

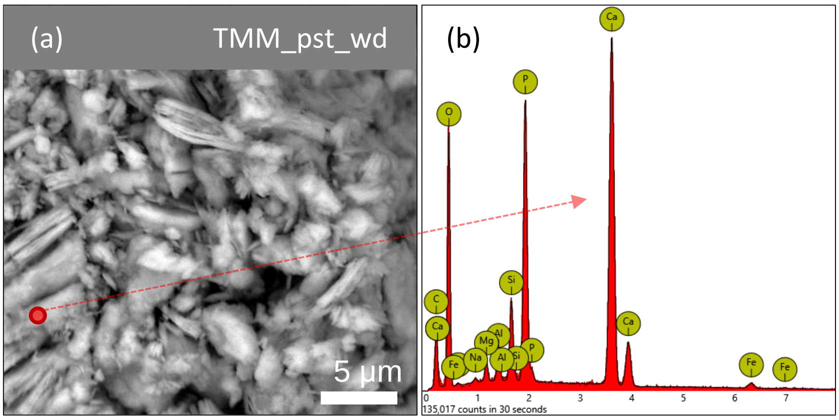


Fig. 9. SEM micrograph (a) and EDX spectrum (b) of bone fragments and ochre powder on the basalt pestle TMM F36516 (Image: B. Melosu, V. Mameli).

Table 4. Semi-quantitative data obtained by SEM-EDX spot analyses. Each row for each sample represents the data for a different spot on the sample. Other elements were detected besides those listed in the table: carbon (C), oxygen (O), fluorine (F), chlorine (Cl), nitrogen (N), sulphur (S), titanium (Ti), and manganese (Mn).

| ***Spot analysis*** | **Atomic concentration (%)** | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Sample\Elements** | **Fe** | **Si** | **Al** | **K** | **P** | **Ca** | **Mg** | **Na** |
| | 2.41 | 24.43 | 7.61 | 2.38 | - | 0.72 | 0.97 | 0.29 |
| | 2.07 | 17.20 | 5.76 | 0.88 | - | 0.89 | 1.42 | 0.20 |
| | 0.93 | 17.52 | 4.72 | 0.54 | - | 0.46 | 0.81 | 0.33 |
| **TMM_soil** | 1.05 | 12.15 | 5.08 | 0.51 | **2.40** | **4.65** | 1.13 | 1.23 |
| | 3.52 | 18.75 | 7.79 | 1.32 | - | 1.15 | 1.27 | 0.43 |
| | 2.14 | 18.35 | 7.44 | 1.12 | 0.55 | 1.22 | 1.20 | 0.19 |
| | 2.21 | 17.97 | 7.03 | 0.93 | - | 0.58 | 1.30 | 0.15 |
| | 40.80 | 6.17 | 3.73 | 0.85 | - | 0.60 | 0.36 | 0.14 |
| | 43.72 | 5.74 | 3.74 | 0.96 | - | 0.59 | - | - |
| **TMM_geo_cm** | 48.93 | 5.08 | 3.18 | 0.65 | - | 0.48 | - | - |
| | 50.39 | 4.85 | 3.24 | 0.88 | - | 0.87 | - | - |
| | 1.69 | 4.21 | 1.38 | 0.23 | - | 0.38 | 0.35 | 0.08 |
| | 2.09 | 14.06 | 4.05 | 0.61 | 0.38 | 1.25 | 0.68 | 0.39 |
| | 1.73 | 6.28 | 2.59 | 0.34 | 0.35 | 1.26 | 0.49 | 0.19 |
| **TMM_gs1_rd** | 2.99 | 17.61 | 6.05 | 0.96 | 0.70 | 1.91 | 1.06 | 0.24 |
| **(TMM 12952)** | 2.20 | 9.60 | 4.45 | 0.45 | 0.41 | 1.54 | 0.71 | 0.46 |
| | 11.41 | 23.54 | 8.55 | 2.57 | 0.45 | 3.57 | 0.77 | 0.35 |
| | 27.42 | 14.97 | 4.88 | 2.44 | 0.45 | 8.11 | 0.40 | 0.10 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 10.88 | 17.52 | 6.32 | 1.09 | **1.63** | **3.14** | 1.09 | 0.19 |
| | 14.96 | 17.28 | 5.95 | 1.37 | **1.85** | **4.71** | 0.81 | 0.46 |
| **TMM_gs2_rd** | 4.67 | 12.85 | 6.37 | 0.71 | **0.52** | **0.84** | 1.12 | 0.48 |
| **(TMM F32971)** | 3.33 | 3.41 | 1.76 | 0.20 | **5.13** | **8.54** | 0.36 | 0.42 |
| | 14.45 | 17.67 | 7.07 | 1.20 | **2.22** | **4.62** | 1.09 | 0.63 |
| | 11.16 | 23.91 | 9.91 | 3.72 | **0.77** | **2.40** | 0.92 | 0.26 |
| | 0.33 | 1.16 | 0.62 | - | **6.22** | **10.95** | 0.54 | 0.30 |
| | 0.43 | 1.98 | 0.92 | - | **5.40** | **8.99** | 0.72 | 0.26 |
| **TMM_pst_wd** | 0.48 | 1.73 | 0.87 | 0.04 | **7.23** | **12.74** | 0.81 | 0.36 |
| **(TMM F36516)** | 0.60 | 2.40 | 1.20 | 0.09 | **8.67** | **14.96** | 1.02 | 0.38 |
| | 0.58 | 1.98 | 0.85 | - | **7.78** | **15.04** | 0.72 | 0.19 |
| | 4.10 | 8.92 | 4.07 | 0.96 | 0.35 | 1.18 | 0.70 | 0.14 |
| | 2.74 | 20.36 | 6.26 | 0.78 | - | 1.09 | 1.21 | 0.08 |
| **TMM_pv1_is_rd** | 2.23 | 11.50 | 5.10 | 0.95 | 0.32 | 0.95 | 0.82 | 0.17 |
| **(TMM1213)** | 2.38 | 12.63 | 4.16 | 0.68 | 0.22 | 0.95 | 0.64 | 0.22 |
| | 5.76 | 17.43 | 7.69 | 1.02 | 0.44 | 1.06 | 1.31 | 0.34 |
| | 1.09 | 20.66 | 8.08 | - | **0.89** | **1.28** | - | 5.97 |
| **TMM_pv1_es_rd** | 6.49 | 5.00 | 2.37 | 0.42 | **3.07** | **4.61** | 0.37 | 0.25 |
| **(TMM1213)** | 1.95 | 5.17 | 2.74 | 0.35 | **7.01** | **11.52** | 0.51 | 0.07 |
| | 1.78 | 2.98 | 1.47 | 0.22 | **4.54** | **8.01** | 0.40 | 0.22 |
| | 15.36 | 11.28 | 4.17 | 0.60 | 0.39 | 0.95 | 0.72 | 0.24 |
| | 3.89 | 13.89 | 6.50 | 0.82 | - | 0.77 | 1.09 | 0.28 |
| | 6.84 | 9.36 | 4.90 | 0.61 | 0.37 | 3.11 | 1.06 | 0.22 |
| | 21.16 | 6.26 | 3.14 | 0.21 | - | 0.15 | 0.35 | - |
| **TMM_pv2_is_rd** | 21.86 | 12.73 | 3.58 | 0.63 | - | 0.67 | 0.42 | - |
| **(TMM F9286)** | 2.83 | 8.62 | 3.38 | 0.31 | 0.14 | 0.38 | 0.41 | - |
| | 3.84 | 6.94 | 2.60 | 0.38 | 0.15 | 0.39 | 0.55 | - |
| | 1.85 | 9.94 | 4.08 | 0.46 | 0.48 | 1.19 | 0.81 | 0.24 |
| | 3.24 | 17.21 | 6.03 | 0.86 | - | 1.08 | 1.24 | 0.27 |
| | 5.94 | 17.44 | 3.33 | 0.43 | **3.09** | **6.61** | 0.60 | 0.24 |
| | 1.45 | 16.06 | 5.56 | 1.20 | **1.61** | **2.94** | 0.72 | 0.72 |
| **TMM_pv3_is_rd** | 2.02 | 13.30 | 4.11 | 0.43 | **0.58** | **1.06** | 0.76 | 0.07 |
| **(TMM12948)** | 1.24 | 18.83 | 6.56 | 1.55 | **-** | **0.68** | 0.49 | 2.47 |
| | 4.54 | 16.06 | 5.79 | 0.71 | **1.02** | **1.85** | 1.12 | 0.04 |
| | 1.93 | 16.56 | 5.43 | 0.82 | | | | |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **TMM_pv4_is_yd (TMM12622)** | **2.70** | 11.84 | 2.98 | 0.36 | **1.17** | **1.39** | 0.82 | 0.09 |
| | **15.46** | 24.22 | 7.51 | 2.05 | **4.32** | **5.40** | 1.33 | 0.16 |
| | **4.69** | 13.80 | 5.06 | 0.86 | **2.16** | **2.53** | 1.31 | 0.17 |
| | **5.68** | 12.91 | 4.90 | 0.70 | **1.82** | **1.98** | 1.25 | 0.07 |
| | **4.79** | 13.96 | 5.01 | 0.73 | **1.58** | **2.13** | 1.20 | 0.26 |

Table 5. Mean values of the atomic concentration ratios Fe/Si+Al, Ca/Si+Al, and P/Si+Al for the samples analysed by SEM-EDX.

| **Sample\Atomic ratios** | **Fe/Si+Al media** | **Ca/Si+Al media** | **P/Si+Al media** |
|---|---|---|---|
| **TMM_soil** | 0.06 | 0.04 | 0.01 |
| **TMM_geo_cm** | **1.43** | **0.02** | ***absent*** |
| **TMM_gs1_rd (TMM 12952)** | **0.22** | 0.08 | 0.01 |
| **TMM_gs2_rd (TMM F32971)** | **0.31** | **0.13** | 0.06 |
| **TMM_pst_wd (TMM F36516)** | **0.02** | **0.39** | **0.22** |
| **TMM_pv1_is_rd (TMM1213)** | 0.11 | 0.03 | 0.01 |
| **TMM_pv1_es_rd (TMM1213)** | 0.09 | **0.20** | **0.12** |
| **TMM_pv2_is_rd (TMM F9286)** | **0.28** | 0.03 | 0.01 |
| **TMM_pv3_is_rd (TMM12948)** | 0.09 | 0.07 | 0.03 |
| **TMM_pv4_is_yd (TMM12622)** | **0.21** | 0.08 | 0.07 |

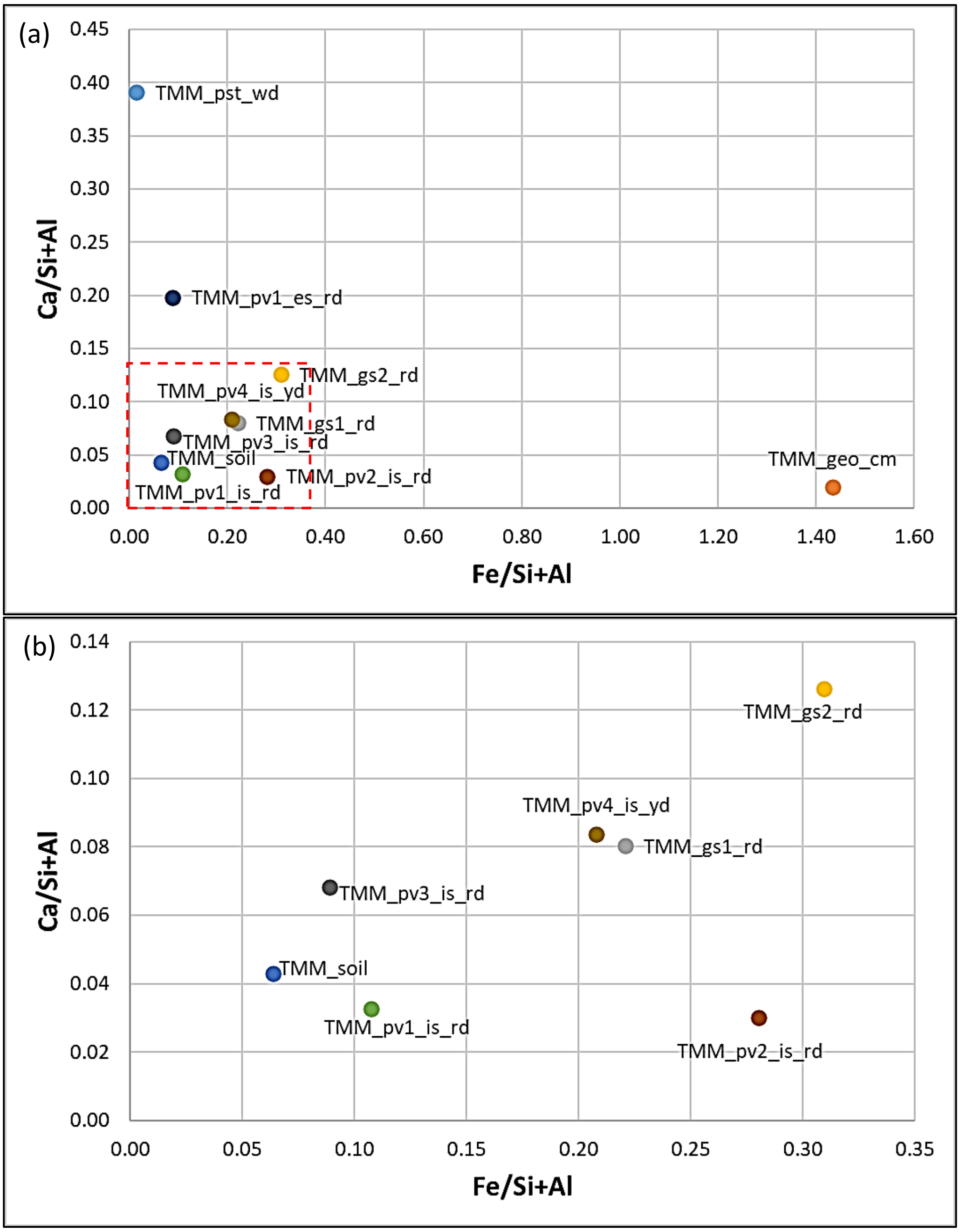


Fig. 10. Scatter plot of the mean atomic concentration ratios Fe/Si+Al versus Ca/Si+Al obtained for the samples analysed by SEM-EDX. (b) is a zoomed area of the (a) graph (Image: V. Mameli).

### *4.2 The use of pottery vessels with residues of colouring materials*

Red deposits with different thickness and distribution on the surfaces were identified on twelve sherds pertaining to five individual vessels (fig. 11-12). As already noted, XRD analyses detected haematite only in red deposits, whereas this mineral was absent from the interior pottery surfaces and the soil sample (table 3). The yellow deposit on the interior surface of another vessel (fig. 12) was not traceable to an iron (hydr)oxide-based pigment, neither to an organic residue, as revealed by XRD, ATR and SEM-EDS analyses (table 3-4, fig. 5-9). Consequently, its origin and function remain unknown. Moreover, no clear evidence of white residues was found on pottery: this suggests a different use for the bone powder processed with lithic tools.

By evaluating the data about morphotypology, morphometry and main technological features of the vessels in relation to the results of use-wear analysis and archaeometric identification of the colour residues, the functional characterisation of the vessels and their role in relation with colouring materials can be elucidated (table 6).

Table 6. Integrated functional analysis of TMM pottery vessels with colour deposits.

| | Vessel/sherd number | Vessel morpho-type | Position of residues | | Appearance and distribution of residues | | Attritions | | Vessel morpho-metry | Technology | Analysis of colour residues: diagnostic features | GC-FID and GC-MS analysis of absorbed organic residues | Assigned functional category |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Ext surf | Int surf | Ext surf | Int surf | Ext surf | Int surf | | | | | |
| 1 | TMM1213 | Bowl with stepped wall | Medium wall | Medium wall | Red deposit, covering | Red deposit, covering | | Horizontal and oblique linear scratches | nd | Fine paste, polished | SEM-EDS: Fe-rich<br>ATR-FTIR: haematite | Not analysed | Processing (and storing?) ochre or ochre-based product |
| 2 | TMM1877 | Bowl with slightly concave base | Lower wall | Lower wall | Red spots, indirect staining | Red deposit, covering | | Horizontal and oblique linear scratches | | Fine paste, burnished | SEM-EDS: Fe-rich<br>ATR-FTIR: haematite<br>XRD: haematite | Concentration < 5 µg/g | Processing (and storing?) ochre or ochre-based product |
| | TMM F9286, TMM12839 | | Lower wall | Lower wall, bottom | Small red spots, indirect staining | Red deposit, covering | Slight peripheral base abrasion | Horizontal and oblique linear scratches | Base Ø 60 mm<br>Vol. min. 430 mL | | SEM-EDS: Fe-rich | | |
| 3 | TMM1974, TMM12645 | Closed deep jar | | Upper wall, rim | | Red subcircular deposits, localised | | | Rim Ø 160 mm<br>Vol min. 500 mL | Medium paste, burnished | Not analysed (small residues) | Concentration < 5 µg/g | Storing ochre powder or ochre-based product |
| 4 | TMM2398 | Flat base (closed deep jar?) | Lower wall | | Very thin layer of red deposit, covering | | | | nd | Fine paste, burnished | Not analysed (thin layer) | Concentration < 5 µg/g | Vessel for storing liquids, exterior surface coated with diluted ochre |
| 5 | TMM12362, TMM2037 | Closed deep jar with stepped wall | Upper wall | | Very thin layer of red deposit, covering | | | | Rim Ø 80 mm<br>Vol min. 1500 mL | Very fine paste, burnished | Not analysed (thin layer) | Concentration < 5 µg/g | Vessel for storing liquids, exterior surface coated with diluted ochre |
| 6 | TMM12622 | Closed deep jar | | Upper wall | | Thin yellow deposit, covering | | | Rim Ø 100 mm<br>Vol. min. 920 mL | Fine paste, burnished | SEM-EDS: Fe-rich<br>XRD: no diagnostic minerals<br>ATR_FTIR: not diagnostic | Concentration < 5 µg/g | Storing undetermined product (colouring material?) |
| 7 | TMM12948, TMM2009 | Bowl with everted rim | Upper wall | Upper wall | Red deposit, covering | Red deposit, covering | | Horizontal and oblique linear scratches | Rim Ø 140 mm<br>Vol. min. 700 mL | Fine paste, polished | SEM-EDS: Fe-rich<br>XRD: haematite | Concentration < 5 µg/g | Processing (and storing?) ochre or ochre-based product |

All but one (TMM1974) vessel with colour residues were fashioned with fine or very fine pastes, and accurately burnished or polished, matching the typical features of most MNB pottery (Fanti 2019). First, in two bowls (TMM1213 and TMM12948), red residues identified as haematite-rich deposits (table 6), covering the interior and exterior surfaces, were associated with horizontal and oblique fine linear scratches on the interior surface (fig. 11). Comparable attrition was found in a bowl with slightly concave base (TMM1877/TMM F9286), possibly pertaining to the same vessel as TMM1213 or TMM12948 but bearing red deposits only on the interior lower wall and bottom, with small spots (indirect staining) on the exterior surface. This use-alteration pattern could be traced back to the effective use of the vessels for processing and mixing ochre, alone or as a main ingredient of composite products, possibly in a liquid medium (water?). The rim diameter of the bowls (ca. 140 mm) and their low restricted mouth enabled handling of the contents (with an estimated total volume of 1 L) with or without tools. The distribution of covering deposits on the upper/medium portions of the exterior surfaces could have been caused by pouring the content. Moreover, the lower portion TMM F9286 had a slight peripheral abrasion on the external base, suggesting movement of the bowl during use activities (fig. 11; Vieugué 2014; Fanti et al. 2018; Fanti 2019; Van Gijn et al. 2020).

Secondly, localised red deposits occurred on the upper interior surface and rim of a closed deep jar (TMM1974/TMM12645; fig. 12), but their small amount did not allow analysis. Based on the appearance of the deposits and the morphotypological and dimensional characteristics of the vessel (table 6), this jar could have been used for storing ochre, in the form of powdered material, not well adhering to the pottery surface, or contents including small amounts of ochre powder as a component.

Finally, the presence of a very thin layer of red deposit only on the exterior surface of sherds, pertaining to closed deep "stepped-wall" jars (TMM12362/TMM2037, TMM2398; fig. 12), was compatible with the application of a highly diluted slip (Gallay et al. 2012; Jones et al. 2019). The application of a red coating is an unconventional choice in the manufacturing process of MNB pottery. Nevertheless, at TMM this behaviour appeared to be strictly limited to closed deep vessels, which are rare and little known in MNB sites (Fanti 2015). Some experimental works investigated the functional efficacy of iron-rich coatings in enhancing performance characteristics of pottery: interestingly, red slips can be useful in reducing absorption of oily contents, but do not significantly help limit water absorption (Longacre et al. 2000; Rueff et al. 2021). The localisation of the coating only on the exterior surface of TMM jars, together with the absence of lipid residues, suggest an aesthetic and/or symbolic purpose, but a functional aim cannot be excluded.

Although lipid traces (mainly fatty acids) were detected in the extracts from six sherds with colour deposits, together with contaminants from plastics, their concentration was lower than the conventional established threshold of 5 µg/g in all the samples (fig. 13; Whelton et al. 2021). In previous work on the pottery from TMM, very low or no lipid amounts were detected in some carinated bowls and jars, besides bowls, pots and big size vessels containing dairy and ruminant adipose fats (Fanti et al. 2018). Interestingly, neither solvent extraction nor acidified methanol extraction provided sufficient release of lipids from TMM samples (fig. 13; Correa-Ascencio, Evershed 2014; Papakosta et al. 2015; Reber 2021). These results can be related to several factors (or a combination of them): the use of vessels for low or no lipidic contents (ochre alone or simply mixed with water), the low porosity of the vessels (Correa-Ascencio, Evershed 2014; Drieu et al. 2019), the absence of a thermic treatment, which can enhance mobilisation of lipids and absorption into the pottery matrix (Evershed 2008b), a complete degradation of the lipidic signal, and, ultimately, a role played by the properties of ochre in limiting the transfer of lipids into the pottery walls (Audouin, Plisson 1982; Rueff et al. 2021).

Therefore, in TMM vessels, the association of animal fat or vegetal oil with ochre, potentially referable to the addition of a liquid/viscous medium to the pigment, was not demonstrated by GC analysis; mixing ochre with a binder cannot be definitively ruled out, notably if it was a proteinic product, such as egg or collagen-based animal glue, or other low-lipid materials, whose biomolecular components would have been rapidly degraded during burial (Rampazzi et al. 2007; Evershed 2008a; Hammann, Cramp 2018; Kozowyk et al. 2020; Miller et al. 2020; Whelton et al. 2021). Nonetheless, the preparation of ochre-based products could have been also conducted simply by adding water (Couraud 1983; Taçon 2012).

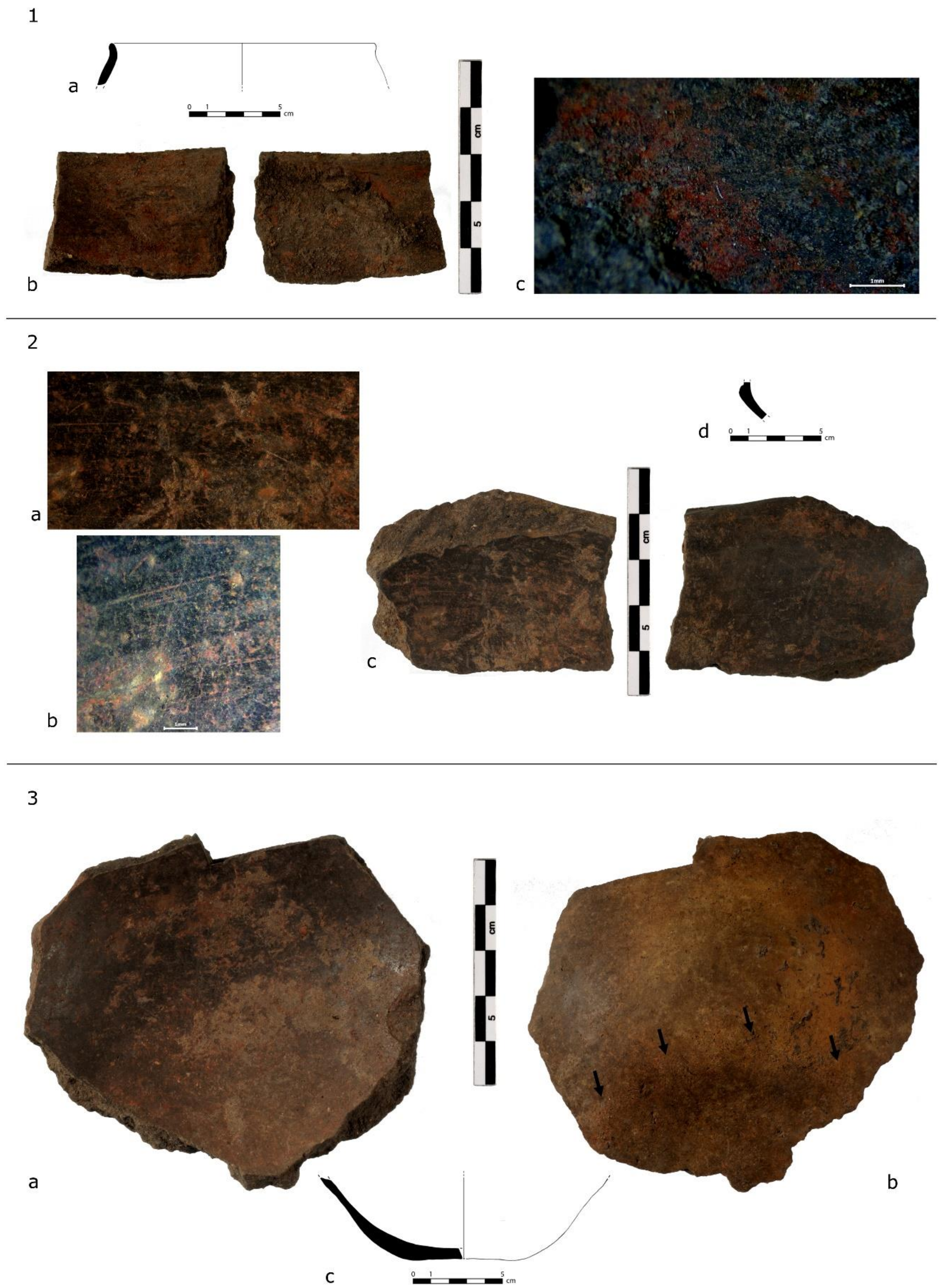


Fig. 11. Pottery vessels with red deposits from the TMM site: 1) TMM12948: a, graphical reconstruction of the vessel morphology, b, red deposits on the interior and exterior surfaces, c, detail of the red deposit (stereomicroscope, 10x); 2) TMM1213: a) attritions (horizontal and oblique linear scratches) and red deposits on the interior surface, b, detail of the linear scratches and red deposits on the interior surface (stereomicroscope, 10x), c, overall view of the sherd with red deposits on the interior and exterior surfaces, d) drawing of the vessel profile; 3) TMM F9286: a, red deposits on the interior surface, b, peripheral base abrasion on the exterior surface, c, graphical reconstruction of the vessel morphology (Image, photo and drawings: L. Fanti).

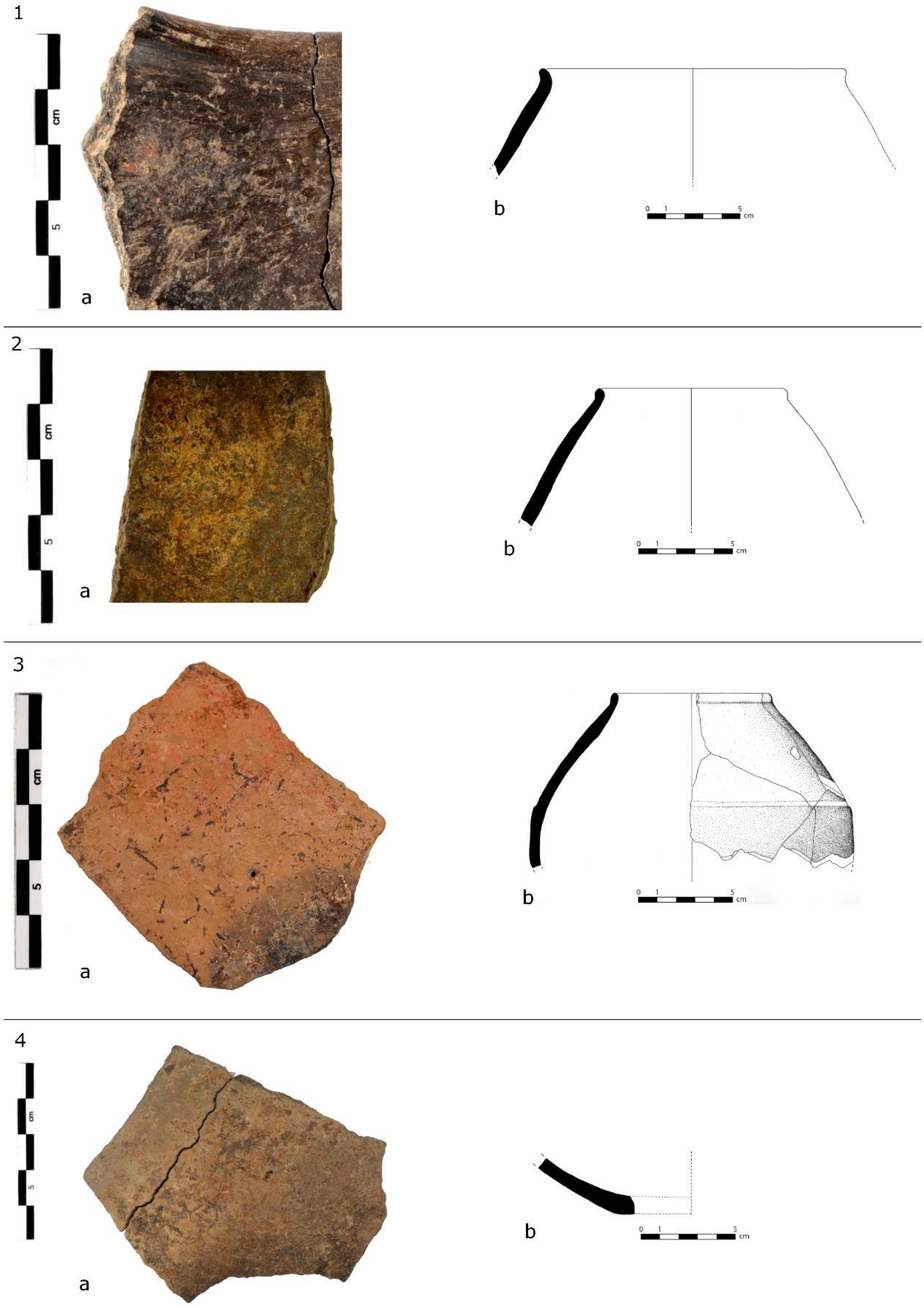


Fig. 12. Pottery vessels with colour deposits from the TMM site: 1) TMM1974: a, deposits of red ochre on the interior surface; b, graphical reconstruction of the vessel morphology; 2) TMM12622: a, yellow deposit on the interior surface; b, graphical reconstruction of the vessel morphology; 3) TMM2037: a, sherd with thin coating of red ochre on the exterior surface; b, reconstructed morphotype (closed deep jar with "stepped wall" TMM4086); 4) TMM2398: a, thin coating of red ochre on the exterior surface; b, drawing of the vessel. (Image, photos, and drawings: L. Fanti).

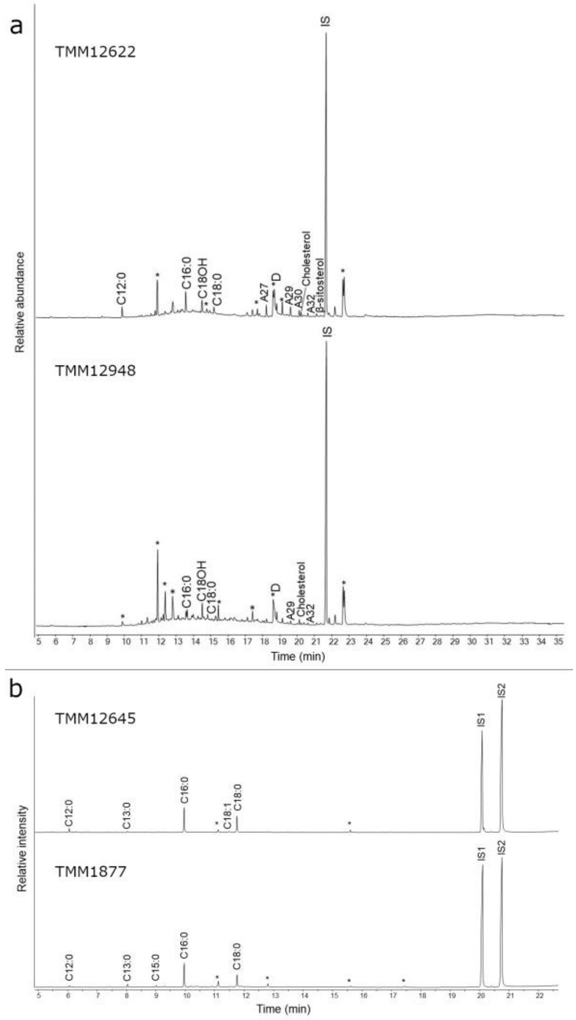


Fig. 13. Partial gas chromatograms of (a) solvent (dichloromethane/methanol) extracted and (b) acidified methanol extracted samples from TMM sherds, showing insufficient lipid residues for interpretation. $C_{n:0}$: saturated fatty acids with n carbon atoms; $C_{n:x}$ unsaturated fatty acids with n carbon atoms; $A_x$: n-alkanes containing x carbon atoms; IS: internal standard ($C_{34}$ n-tetratriacontane), IS1: $C_{34}$ n-tetratriacontane; IS2: $C_{36}$ n-hexatriacontane; *: plasticisers (phthalates), *D: 13-docosenamide (Image: L. Fanti).

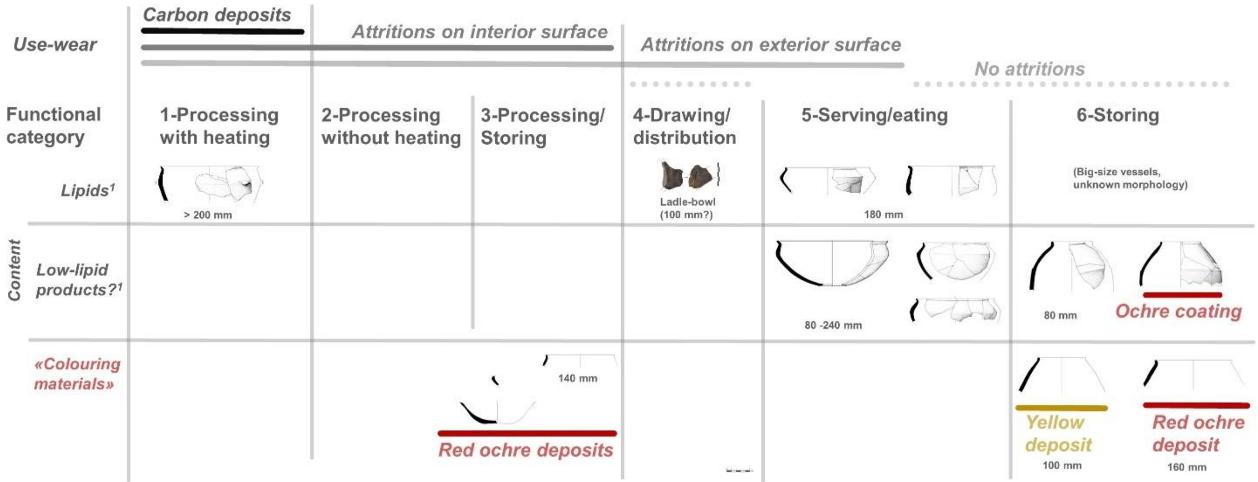


Fig. 14. Overall functional structure of the pottery assemblage from TMM site, defined by combining use-wear analysis and data on contents, including vessels associated with colouring materials. 1: data from Fanti et al. 2018. Measures in mm refer to the rim diameter range in the different functional categories (Image, photo and drawings: L. Fanti).

It is worth considering the use of vessels with colouring materials in the framework of the overall functional structure of the pottery assemblage from TMM (fig. 14; Fanti et al. 2018; Fanti 2019). Pottery appears to be involved both with the management of haematite-based materials (processing, storing) and their final use (coating of vessel surface). The vessels seem to have been chosen for these purposes according to their morphology and morphometry: closed deep jars for storing (rim Ø: 80-160 mm), low restricted and shallow bowls for processing/storing (rim Ø: 140 mm), in front of a wider range of morphologies and dimensions of bowls (rim Ø: 80-240 mm) used for serving/eating fats and low-lipid products (Fanti et al. 2018, Fanti 2019). No pots, ladle-bowls nor big-size vessels were (re)used for colouring materials.

Overall, the specimens with ochre deposits correspond to 4% of the total number of individual vessels (138). Consequently, the role of pottery in handling haematite-rich products could appear to have been marginal: nonetheless, this could be also related with the value of ochre, as a precious or symbolically invested material, involved only in infrequent activities, and/or in activities requiring low amount of product each time. As regards the possible multifunctionality or reuse of the vessels, the absence of absorbed lipids into the pottery walls might indicate that these bowls and jars have been previously utilised also to serve, eat or store low-lipid contents, or they were only used for the specific purpose of containing ochre. However, they do not seem to have been frequently moved, considering the low abrasion of the external bases (Vieugué 2014; Fanti 2019).

In the TMM site, the occurrence of ochre as residue of the original vessels contents stimulates further discussion on the effective use of ochre-based products. In addition to its application as a red pigment, ochre might have been involved in various technical activities, the best known of which is hide processing, where ochre can be exploited for its abrasive properties but can also take effect as preservative agent (Audouin, Plisson 1982; Rifkin 2011). Moreover, ochre can be used as an abrasive addictive in polishing wood or bone artefacts (Domingo et al. 2012) or as a charge in the production of hafting glues (Wadley 2005; Kozowyk et al. 2020). Nevertheless, no evidence of these kinds of uses has been identified in TMM nor in other MNB contexts thus far. As highlighted by many ethnoarchaeological studies, besides culinary functions, pottery vessels are sometimes used as containers for storing raw materials for technical activities (Arnold 1985; Deal 2011), for preparing and storing products for body care and hygiene, as well as medical remedies (Gosselain, Van Berg 1992; Diop 2000; Insoll 2011; Gallay et al. 2012; Grillo 2014; Huysecom et al. 2017). The pottery vessels from TMM could have been also used to prepare and store not only ochre as pigment or abrasive/prophylactic additive for technical activities, but also specific haematite-based products for body decoration (Dater-Wolf et al. 2021) or for topical application, *e.g.*, to treat wounds or diseases (Salomon 2009, with references therein; Teklay et al. 2022). As a matter of fact, haematite/red ochre was a component of ancient and traditional medicines worldwide (Shemluck 1982; De Vos 2010; Kadioglu et al. 2016; Fazil, Nikhat 2020; Knapp et al. 2021; Russell et al. 2021; Li et al. 2022). In this sphere, an intriguing possibility for the presence of residues in pottery vessels could be the consumption of ochre, alone or mixed with other edible components in medicinal remedies (Russell et al. 2021). Interestingly, eating of ochre (as well as different kinds of clays) for magical-medical purposes, a behaviour imbued of deep symbolic implications, is attested in some ethnographic contexts (Teklay et al. 2022). Moreover, ochre can be added to food as an antimicrobial agent (Couraud 1983, Taçon 2012 p. 34 with references therein). Although similar uses cannot be directly proved at TMM and, generally, in prehistoric contexts, they cannot be definitively ruled out, especially in household settlements. A connection between the activities carried out at TMM and the decoration of special items or ritual/funerary practices performed elsewhere is equally another possibility.

By combining all the data obtained through our integrated analysis, a first picture of the technology of ochre in the MNB open-air settlement of TMM, including the role assigned in that to pottery vessels and its possible actual uses, can be finally proposed (fig. 15).

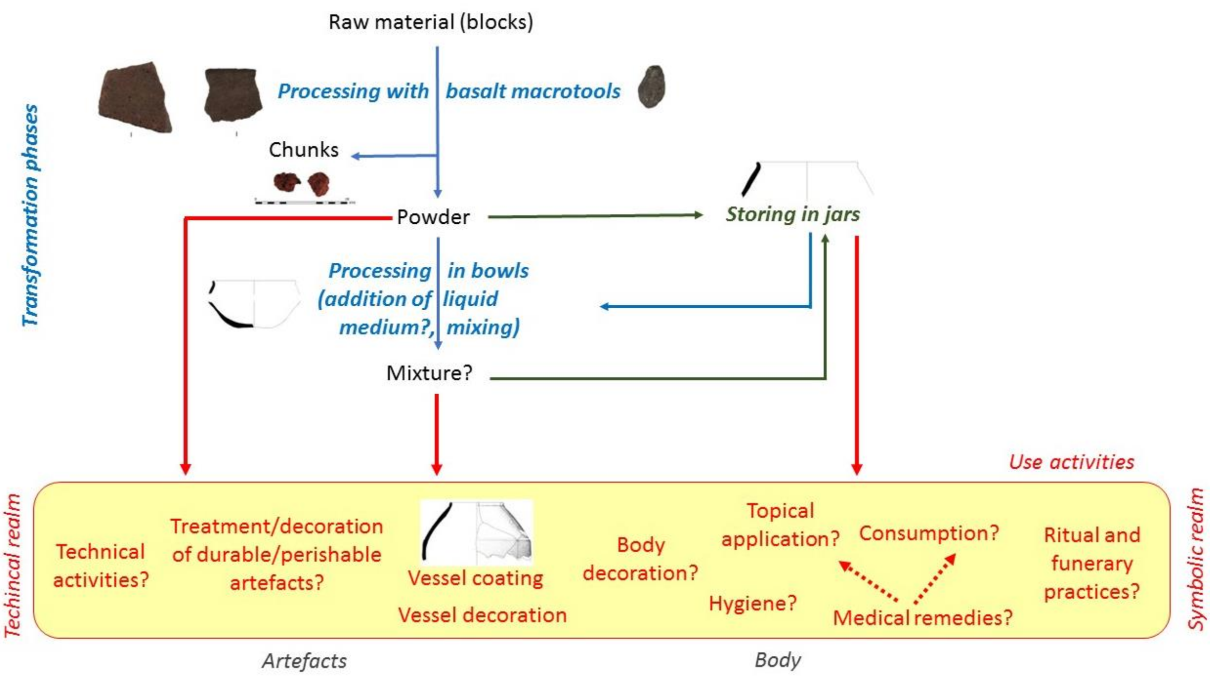


Fig. 15. Partial *chaîne opératoire* of red ochre in the TMM site, with attested or possible uses of haematite-based products. Black: state of material; blue: transformation phases; green: storing phases; red: use activities (Photos: B. Melosu; image and drawings: L. Fanti).

## 5. Conclusions

In this study, the technology of colouring materials, particularly red ochre, in Sardinia during the Middle Neolithic has been addressed through an interdisciplinary approach, combining functional analysis of pottery and study of lithic macro-tools together with a multi-technique archaeometric identification of the elemental, structural and mineralogical composition of geomaterials and colour residues on artefacts. Based on previous data, the processing and use of (not analysed) "red ochre" was mainly related to burials and, secondarily, to decoration. This work broadened knowledge about the technology of colouring material, integrated in the activities carried out by MNB societies in an open-air household settlement. Our results demonstrated the processing and use of haematite-rich ochre as the exclusive red colour-producing material, occurring both as deposits on artefacts and residual chunks, from *in situ* transformation. Additional analyses are needed to assess whether all samples come from a single or multiple ochre sources, thus resulting from a unidirectional or a composite network of supply of raw materials. Further mandatory step of the research should include systematic investigation into the provenance of geomaterials (Lugliè 2020b).

The integrated analysis showed the role of basalt macro-tools (grinding slabs, pestle) and pottery (bowls, jars), used for handling ochre. Significantly, basalt tools were associated with red ochre from Early until Late Neolithic in Sardinian sites (Agosti et al. 1980; Trump 1983; Santoni 2019): this could reflect a long-lasting recognised effectiveness of this igneous rock for processing activities related to colouring materials. At TMM, ochre was used as pigment, applied on the exterior surface of few jars, probably for aesthetic and/or symbolic purposes. The analysis of use alteration associated with haematite-rich deposits in some shallow bowls and a closed deep jar points to the selection of these morphotypes in processing and storing ochre or haematite-based products; however, mixing with organic components was not demonstrated. As suggested by ethnoarchaeological, experimental and ethnopharmacological studies, the actual uses of red ochre in household settlements could encompass various technical applications (as drying, abrasive, preservative agent), and possibly medicinal purposes, besides the colouring function for decorating durable or perishable artefacts (and body?) and its symbolic connection with ritual or funerary contexts.

Diachronically, the exploitation of colouring materials in Sardinia seems to grow progressively throughout the Early and Middle Neolithic (fig. 1), culminating in the widespread adoption and utilisation of pigments in pottery decoration and wall paintings in the *Domus de Janas* hypogeal tombs from the Late Neolithic (4th millennium cal BCE; Rampazzi et al. 2002; Tanda 2003; Tanda et al. 2003; Rampazzi et al. 2007; Melosu 2020). New systematic investigation is necessary to highlight continuity or crucial innovation points in the management systems of colouring materials (selection of geomaterials, procurement, processing, handling and storing methods, role of pottery vessels), in relation with other evidence of cultural shifts in the different Neolithic phases.

At this stage of the research, the general lack of techno-functional studies on colouring materials limits a deeper interpretation of the results from the TMM site in a wider perspective. The most interesting chrono-cultural sphere for comparison can be found in the central-northern peninsular regions of Italy: various archaeological clues (pottery decorations, obsidian circulation, ornaments, and particular funerary customs, *i.e.* cremation) showed the bidirectional relationships and influences between MNB groups and Square Mouth pottery (SMP) societies (Lugliè et al. 2019; Lugliè 2020a-b; Paba et al. 2021). Specifically, pigments were involved in pottery decoration, notably in the SMP "meander-spiral" style, and related to the symbolic sphere (funerary sites, anthropomorphic figurines); moreover, pottery vessels were used for storing red ochre in several

SMP contexts (Bernabò Brea et al. 2006; Bernabò Brea, Mazzieri 2011). The practices involving colouring materials would complement the evidence on the movements of objects, people, and ideas from and towards the islands across the Tyrrhenian Sea during the 5th millennium.

Nevertheless, only the multiplication of integrated functional and archaeometric approaches in other Mediterranean regions could lead to better define analogies and identify divergences among technical traditions and symbolic expressions of different Neolithic groups, and, with regard to Sardinia and other insular regions (*e.g.*, Corsica), to elucidate the role played by seafaring contacts in shaping and diffusing techniques and behaviours related to "colouring materials". Hopefully, further research should evaluate the use of ochre for its multiple properties (Pradeau 2015), particularly when associated with pottery vessels, both from a regional and interregional viewpoint, exploiting the many reported remains bearing colour residues from Neolithic contexts within the Western Mediterranean area.

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## AUTHOR'S CONTRIBUTION

All the authors equally contributed to conceptualisation, methodology, investigation, data validation. Laura Fanti carried out morfotypological and functional analysis of the TMM pottery collection, GC-FID and GC-MS analyses. Barbara Melosu studied lithic artefacts and ochre samples from all archaeological materials and performed SEM-EDS and ATR-FTIR analysis. Valentina Mameli and Carla Cannas conducted ATR-FTIR and XRD analyses.

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## REFERENCES


Agosti F., Biagi P., Castelletti L., Cremaschi M., Germanà F. 1980, La grotta Rifugio di Oliena (Nuoro): caverna ossario neolitica, *Rivista di Scienze Preistoriche* XXXV: 75-124.

Angeli L., Arias C., Cristoforetti G., Fabbri C., Legnaioli S., Palleschi V., Radi G., Salvetti A., Tognoni E. 2006, Spectroscopic techniques applied to the study of Italian painted Neolithic potteries, *Laser Chemistry*, 61607.

Angeli L., Arias C., Fabbri C., Radi G., Cristoforetti G., Legnaioli S., Palleschi V., Salvetti A., Tognoni E. 2011, L'impiego dell'ossido di manganese nelle ceramiche dipinte del Neolitico, in *Il Fucino e le aree limitrofe nell'antichità. Atti del III Convegno di Archeologia in ricordo di Walter Cianciusi, Castello Orsini, Avezzano, 13-15 novembre 2009*, Avezzano, Archeoclub d'Italia-Sezione della Marsica: 564-566.

Angeli L., Legnaioli S., Fabbri C., Lorenzetti G., Guilaine J., Palleschi V., Radi G. 2018, Analysis of Serra d'Alto *figuline* pottery (Matera, Italy): Characterisation of the dark decorations using XRF, *Microchemical Journal* 137: 174-180.

Angeli L., Brunetti A., Legnaioli S., Fabbri C., Campanella B., Lorenzetti G., Pagnotta S., Poggialini F., Palleschi V., Radi G. 2019, Analysis of the middle Neolithic trichrome pottery: Characterization of the decoration using X-Ray fluorescence and Raman spectroscopy, *Journal of Archaeological Science: Reports* 24: 192-197.

Antona A. 1998, Le statuette di dea madre nei contesti prenuragici: alcune considerazioni. *In* Balmuth M. S., Tykot R. H. (eds.), *Sardinian and Aegean Chronology: Towards the Resolution of Relative and Absolute Dating in the Mediterranean. Proceedings of the International Colloquium 'Sardinian Stratigraphy and Mediterranean Chronology', Tufts University, Medford, Massachusetts, March 17-19, 1995,* Oxford Oxbow Books: 111-119.

Antona A. 2003, Il megalitismo funerario in Gallura. Alcune considerazioni sulla necropoli di Li Muri, *Rivista di Scienze Preistoriche* LIII: 359-372.

Antona A. 2013, *Arzachena. Pietre senza tempo*, Sassari, Carlo Delfino editore.

Antona A. 2020, La necropoli di Li Muri, In Cossu T., Lugliè C. (eds.), *La preistoria in Sardegna. Il tempo delle comunità umane dal X al II millennio a.C.*, Nuoro, Ilisso: 114-117.

Armetta F., Giuffrida D. Martinelli M. C., Mollica Nardo V., Saladino M. L., Ponterio R. C. 2023, Non-invasive investigation on pigments of the Aeolian Islands Neolithic pottery, *Materials Letters* 336: 133854.

Arnold D. E. 1985, *Ceramic Theory and Cultural Process,* Cambridge, Cambridge University Press.

Atzeni E. 1975, Nuovi idoli della Sardegna prenuragica, *Studi Sardi* XXIII: 3-51.

Audouin F., Plisson H. 1982, Les ocres et leurs témoins au Paléolithique en France : enquête et expériences sur leur validité archéologique, *Cahiers du Centre de Recherches Préhistoriques* 8: 33-80.

Barra A., Grifoni Cremonesi R., Mallegni F., Piancastelli M., Vitiello A., Wilkens B. 1990, La Grotta Continenza di Trasacco. I livelli a ceramiche, *Rivista di Scienze Preistoriche* 42: 31-100.

Bernabeu Aubán, J., Molina, L., García-Borja, P., 2008. El color en las producciones ceramicas del Neolítico antiguo. *Veleia* 24-25: 655-668.

Bernabò Brea M., Salvadei L., Maffi M., Mazzieri P., Mutti A., Sandias M. 2006, Le necropoli dei Vasi a Bocca Quadrata dell'Emilia occidentale: rapporti con gli abitati, rituali, corredi, dati antropologici, In Pessina A., Visentini P. (eds), *Preistoria dell'Italia settentrionale. Studi in ricordo di Bernardino Bagolini*. Atti del Convegno, Udine, 23-24 settembre 2005: 169-186.

Bernabò Brea M., Mazzieri P. 2011, Stilemi decorativi e significati simbolici nella decorazione vascolare VBQ, *Preistoria Alpina* 46: 7-19.

Bernabò Brea M., Mazzieri P. 2014, Osservazioni sulla sfera rituale del mondo VBQ in base ai dati forniti dagli insediamenti dell'Emilia occidentale, *Rivista di Studi Liguri* LXXVII-LXXIX: 315-321.

Camps G. 1988, *Préhistoire d'une île. Les origines de la Corse*, Paris, Editions Errance.

Cassano S., Manfredini A., Carboni G., Marconi N., Muntoni I. 2003, Il villaggio neolitico di Masseria Candelaro (FG): una premessa archeologica, In *Atti della XXX Riunione Scientifica, Le comunità della Preistoria italiana. Studi e ricerche sul Neolitico e le età dei metalli*, Castello di Lipari, Chiesa di S. Caterina, 2 -7 giugno 2000, Volume II, Firenze, Istituto Italiano di Preistoria e Protostoria: 813-817.

Cipolloni-Sampò M. 1982, Gli scavi nel villaggio neolitico di Rendina (1970-1976): relazione preliminare, *Origini: preistoria e protostoria delle civiltà antiche* 11: 183-323.

Colombo M. 2006, Uno strumento in osso decorato con cinabro dal villaggio neolitico di Catignano (Pescara). Analisi preliminare, In *Atti della XXXIX Riunione scientifica, Materie prime e scambi nella preistoria italiana, Firenze 25-27 novembre 2004*, Vol. II, Firenze, Istituto Italiano di Preistoria e Protostoria: 979-982.

Colombo M. 2012, Idoletti fittili antropomorfi e zoomorfi dal villaggio neolitico di Catignano (PE): studio tipologico e proposta interpretativa, *Preistoria Alpina* 46: 167-173.

Colombo M., Boschian G. 2009, High-technology manufacturing of 5th millennium B.C. Pottery in Italy, *Materials and Manufacturing Processes* 24: 928-933.

Cornell R.M., Schwertmann U. 1996, *The Iron Oxides*, Weinheim, Wiley-VCH Verlag GmbH & Co. KGaA, FRG.

Correo-Ascencio M., Evershed R. P. 2014, High throughput screening of organic residues in archaeological potsherds using direct acidified methanol extraction, *Analytical Methods* 6: 1330-1340.

Couraud C. 1983, Pour une étude méthodologique des colorants préhistoriques, *Bulletin de la Société Préhistorique Française* 80: 104-110.

Dal Rì C., Pedrotti A., Volpin S. 2001, La Vela (TN), excavation campaigns 1987-1988. Mineralogical and chemical analysis carried out on red-pigmented finds in grave n. 3, *Preistoria Alpina* 33: 49-52.

Dater-Wolf A., Peres T. M., Karacic S. 2021, Ancient native American bone tattooing tools and pigments: evidence from central Tennessee, *Journal of Archaeological Science: Reports* 37: 103002.

Daura J., Sanz M., Oms F. X., Pedro M., Martínez P., Mendiela S., Poveda M. O., Gibaja J. F., Mozota M.,Alonso-Eguíluz M., Albert R. M., Allué E., Bañuls-Cardona S., López-García J. M., Santos Arévalo F. J., Fullola J. M. 2019, Deciphering Neolithic activities from a Cardial burial site (Cova Bonica) on the western Mediterranean coast, *Journal of Archaeological Science: Reports* 23: 324-347.

Dayet L. 2021, Invasive and non-invasive analyses of ochre and iron-based pigment raw materials: a methodological perspective, *Minerals* 11: 2010.

Deal M. 2011, Microtradition and Agency in Domestic Pottery Production: an Ethnoarchaeological Perspective, In Scarcella S. (ed.), *Archaeological Ceramics: A Review of Current Research*, BAR International Series 2193, Oxford, Archaeopress: 147-159.

Debels P., Galant P., Vernant P. 2023, Drinking in the dark. A new method to distinguish use-alteration from natural alteration on Neolithic pots and evidence of acid liquid storage in karstic cave contexts, *Journal of Archaeological Science* 150: 105706.

Defrasne C., Chalmin E., Bellot-Gurlet L., Thirault E., André G. 2019, From archaeological layers to schematic rock art? Integrated study of the Neolithic pigments and pigmented rocks at the Rocher du Château (Western Alps, Savoie, France), *Archaeological and Anthropological Sciences* 11: 6065-6091.

De Pascale A. 2014, Le pintadere neolitiche nelle collezioni del Museo Archeologico del Finale, *Rivista di Studi Liguri* LXXVII-LXXIX: 203-210.

De Vos 2010, European materia medica in historical texts: longevity of a tradition and implications for future use, *Journal of Ethnopharmacology* 132: 28-47.

Diop B. 2000, Recherches ethnoarchéologiques sur la céramique au Sénégal, production artisanale et consommation domestique, In Benoît P., Fluzin P., Pétrequin P., Thiriot J. (eds.), *Arts du feu et productions artisanales. XX[e] Rencontres internationales d'Archéologie et Histoire d'Antibes, 21-23 octobre 1999,* Antibes, Éditions APDCA : 261-285.

Domingo I., Chieli A, 2021, Characterizing the pigments and paints of prehistoric artists, *Archaeological and Anthropological Sciences* 13: 196.

Domingo I., García-Borja P., Roldán C. 2012, Identification, processing and use of red pigments (hematite and cinnabar) in the Valencian Early Neolithic (Spain), *Archaeometry* 54: 868-892.

Drieu L., Lepère C., Regert M. 2019, The missing step of *chaîne opératoire*: considering post-firing treatments on ceramic vessels using macro- and microscopic observation and molecular analysis, *Journal of Archaeological Method and Theory* 27: 302-326.

Drieu, L., Lucquin A., Cassard L., Sorin S., Craig O. E., Binder D., Regert M. 2020, A Neolithic without diary? Chemical evidence from the content of ceramics from the Pendimoun rock-shelter (Castellar, France, 5750-5150 BCE), *Journal of Archaeological Science: Reports* 35: 102682.

Evershed R. P. 2008a, Organic residue analysis in archaeology: the archaeological biomarker revolution, *Archaeometry* 50: 895-924.

Evershed R. P. 2008b, Experimental approaches to the interpretation of absorbed organic residues in archaeological ceramics, *World Archaeology* 40: 26-47.

Fabbri B., Gualtieri S., Lorenzi R. 2013, Preliminary archaeometric study of the Neolithic pottery from the "Le Grottelline" site (Spinazzola, Italy), *Archaeological and Anthropological Sciences* 5: 235-243.

Fanti L. 2015, *La fonction des récipients céramiques dans les sociétés du Néolithique Moyen B (4500-4000 cal BC) en Sardaigne centre-occidentale (Italie). Indices fonctionnels, économiques, interculturels à partir de l'analyse des caractéristiques morphométriques, des résidus organiques et des traces d'usure des poteries*, PhD thesis, Université Nice Sophia Antipolis, France.

Fanti L. 2019. Beyond the surface. Functional analysis of pottery and its application to middle Neolithic "*San Ciriaco*" vessels (5th millennium cal BC, Sardinia, Italy)", *Rivista di Scienze Preistoriche* LXIX: 23-55.

Fanti L. 2020. Contenere, cuocere, conservare: l'uso delle ceramiche, In Cossu T., Lugliè C. (eds.), *La preistoria in Sardegna. Il tempo delle comunità umane dal X al II millennio a.C.*, Nuoro, Ilisso: 118-121.

Fanti L., Drieu L., Mazuy A., Blasco T., Lugliè C., Regert M. 2018, The role of pottery in Middle Neolithic societies of western Mediterranean (Sardinia, Italy, 4500-4000 cal BC) revealed through an integrated morphometric, use-wear, biomolecular and isotopic approach, *Journal of Archaeological Science* 93: 110-128.

Fazil M., Nikhat S. 2020, Topical medicines for wound healing: a systematic review of Unani literature with recent advances, *Journal of Ethnopharmacology* 257: 112878.

Fenu P. 2013, La Grotta Su Coloru a Laerru, In Usai L. (ed), *Memorie dal sottosuolo. Scoperte archeologiche nella Sardegna centro-settentrionale. Catalogo della mostra, Sassari, Museo Nazionale "Giovanni Antonio Sanna", Padiglione Clemente, febbraio 2011 - aprile 2013*, Quartucciu, Scuola Sarda Editrice: 37-45.

Fenu P. 2017, La Sardegna preistorica: Catalogo dei materiali, 62 – Tazza carenata. In *Corpora delle antichità della Sardegna. La Sardegna preistorica. Storia, materiali, monumenti*, Firenze, Carlo Delfino Editore: 277.

Ferrarese Ceruti M. L. 1992, Statuine di Dea Madre da Torralba e Ozieri (Sassari), In Sardinia Antiqua. Studi in onore di Piero Meloni in occasione del suo settantesimo compleanno, Cagliari, Edizioni della Torre: 63-74.

Ferrari A., Pessina A. 2012, Oggetti d’arte e di culto dal sito neolitico di Sammardenchia (Pozzuolo del Friuli, Udine), *Preistoria Alpina* 46: 175-184.

Foschi A. 1982, Il Neolitico antico della Grotta Sa Korona di Monte Majore (Thiesi, Sassari). Nota preliminare, In *Le Néolithique Ancien Mediterranéen. Actes du colloque international de préhistoire, 6-8 juin 1981, Montpellier*, Sète, Fédération archéologique de l’Hérault: 339-346.

Foschi Nieddu A. 1987, La grotta Sa Korona di Monte Majore (Thiesi, SS). Primi risultati dello scavo 1980. In *Atti della XXVI Riunione Scientifica dell’Istituto Italiano di Preistoria e Protostoria “Il Neolitico in Italia” (7-10 Novembre 1985)*, Firenze, Istituto Italiano di Preistoria e Protostoria: 859-870.

Foschi Nieddu A. 2002, Grotta Sa Korona, *In* Fugazzola Delpino M. A., Pessina A., Tiné V. (eds), *Le ceramiche impresse nel Neolitico antico. Italia e Mediterraneo*, Roma, Soprintendenza Speciale al Museo Nazionale Preistorico Etnografico "L. Pigorini": 433-439.

Fugazzola Delpino M. A. 2002, La Marmotta (Lazio), In Fugazzola Delpino M. A., Pessina A., Tiné V. (eds), *Le ceramiche impresse nel Neolitico antico. Italia e Mediterraneo*, Roma, Istituto Poligrafico e Zecca dello Stato: 373-395.

Fugazzola Delpino M. A., Tinè V. 2002-2003, Le statuine fittili femminili del Neolitico italiano. Iconografia e contesto culturale, *Bullettino di Paletnologia Italiana* 93-94: 19-51.

Gajić-Kvaščev M., Marić Stojanović M, Šmit Ž., Kantarelou V., Karydas A. G., Šljivar D., Milovanovič D., Andrić V. 2012, New evidence for the use of cinnabar as a colouring pigment in the Vinča culture, *Journal of Archaeological Science* 39: 1025-1033.

Gallay A., Huysecom E., Mayor A., Gelbert A. 2012, *Potières du Sahel : à la découverte des traditions céramiques de la boucle du Niger (Mali),* Gollion, Infolio.

García-Borja P., Domingo Sanz I., Roldán García C., Verdasco Cebrían C., Ferrero Calabuig J., Jardón Giner P., Bernabeu Aubán J. 2004, Aproximación al uso de la materia colorante en la Cova de l’Or, *Recerques del Museu d’Alcoi* 13: 35-52.

García-Borja P., Domingo I., Roldán C. 2006, Nuevos datos sobre el uso de materia colorante durante el Neolítico antiguo en las comarcas centrales valencianas, *Saguntum* 38: 49-60.

Ge W., Liu L., Huang W., Tao S., Hou X., Yin X., Wu Y. 2021, Neolithic bone meal with acorn: analyses on crusts in pottery bowls from 7000 BP Hemudu, China, *International Journal of Osteoarchaeology* 31: 1118-1134.

Germanà F., Mallegni F., Pompeis (de) C., Ronco D. 1990, Il villaggio neolitico di Villa Badessa (Pescara): aspetti paletnologici, antropologici e paleopatologici, *Atti della Società Toscana di Scienze Naturali, Memorie, Serie A* 97: 271-310.

Giustetto R., Berruto G., Diana E., Costa E. 2013, Decorated prehistoric pottery from Castello di Annone (Piedmont, Italy): archaeometric study and pilot comparison with coeval analogous finds, *Journal of Archaeological Science* 40: 4249-4263.

Gliozzo E. 2021, Pigments – Mercury-based red (cinnabar-vermilion) and white (calomel) and their degradation products, *Archaeological and Anthropological Sciences* 13: 210.

Gorgoglione M., Laviano R., Muntoni I. M. 2012, Simbolismo e arte nella Puglia meridionale dalla fine del VI al IV millennio a. C., *Preistoria Alpina* 46: 159-166.

Gosselain, O. P., Van Berg P.-L. 1992, Style, individualité et taxonomie chez les potières Bafia du Cameroun, *Bulletin du Centre Genevois d'Anthropologie* 3: 99-114.

Graziosi P. 1973, *L'arte preistorica in Italia*, Firenze, Sansoni.

Graziosi P. 1980, *Le pitture preistoriche della Grotta di Porto Badisco*, Firenze, Giunti Martello.

Grifoni R. 1967, La Grotta dell'Orso di Sarteano, *Origini: preistoria e protostoria delle civiltà antiche* 1: 53-115.

Grifoni Cremonesi R., Radmilli A. 2001, La Grotta Patrizi al Sasso di Furbara (Cerveteri, Roma), *Bullettino di Paletnologia Italiana* 91-92: 63-117.

Grifoni Cremonesi R., Pedrotti A. 2012, L'arte del Neolitico in Italia: stato della ricerca e nuove acquisizioni, *Preistoria Alpina* 46: 115-131.

Grillo K. M. 2014, Pastoralism and pottery use: an ethnoarchaeological study in Samburu, Kenya, *African Archaeological Review* 31: 105-130.

Guerri M. 1988a, Porto Badisco (Otranto, Prov. di Lecce), Notiziario, *Rivista di Scienze Preistoriche* XLI: 385.

Guerri M. 1988b, Grotta Cosma (S. Cesarea Terme, Prov. di Lecce), Notiziario, *Rivista di Scienze Preistoriche* XLI: 420-421.

Guilaine J. 1996, Protomégalithisme, rites funéraires et mobiliers de prestige néolithiques en Méditerranée occidentale, *Complutum Extra* 6 : 123-140.

Hammann S., Cramp L. J. E. 2018, Towards the detection of dietary cereal processing through absorbed lipid biomarkers in archaeological pottery, *Journal of Archaeological Science* 93: 74-81.

Hamon C., Billard C., Bosquet D., Constantin C., Jadin I. 2016, Usages et transformation de l'hématite dans le Néolithique ancien d'Europe du Nord-Ouest, *Anthropologica & Praehistorica* 125: 45-61.

Heron C., Evershed R. P., Goad L.J. 1991, Effects of migration of soil lipids on organic residues associated with buried potsherds, *Journal of Archaeological Science* 18: 641-659.

Hodgskiss T. 2010, Identifying grinding, scoring and rubbing use-wear on experimental ochre pieces, *Journal of Archaeological Science* 37: 3344-3358.

Horn K. R. 2018, Time takes its toll: Detection of organic binder media in ochre paints with visible near-infrared and short-wave infrared reflectance spectroscopy, *Journal of Archaeological Science: Reports* 21: 10-20.

Howard C. D. 2010, On seeing red: iron oxide on excavated artefacts, *Plains Anthropologist* 55: 83-85.

Huysecom E. et al. 2017, Milieu et techniques dans la Falémé (Sénégal oriental) et sondages au royaume d'Issiny (Côte d'Ivoire) : résultats de la 19ème année du programme « Peuplement humain et paléoenvironnement en Afrique », *SLSA Rapport annuel* 2016: 109-208.

Insoll T. 2011, Substance and materiality? The archaeology of Talensi medicine shrines and medicinal practices, *Anthropology & Medicine* 18: 181-203.

Jones R., Towers R., Card N., Odling N. 2019, Analysis of coloured Grooved Ware sherds from the Ness of Brodgar, Orkney, *Journal of Archaeological Science: Reports* 28: 102014.

Kadioglu O., Jacob S., Bohnert S., Nass J., Saeed M. E. M., Khalid H., Merfort I., Thines E., Pommerening T., Efferth T. 2016, Evaluating ancient Egyptian prescriptions today: anti-inflammatory activity of *Ziziphus spina-christi*, *Phytomedicine* 23: 293-306.

Knapp C. W., Christidis G. E., Venieri D., Gounaki I., Gibney-Vamvakari J., Stillings M., Photos-Jones 2021, The ecology and bioactivity of some Greco-Roman medicinal minerals: the case of Melos earth pigments, *Archaeological and Anthropological Sciences* 13: 166.

Kozowyk P. R. B, van Gijn A. L., Langejan G. H. J 2020, Understanding preservation and identification biases of adhesives through experimentation, *Archaeological and Anthropological Sciences* 12: 209.

Li X., Wu L., Wu R., Sun M., Fu K., Kuang T., Wang Z. 2022, Comparison of medicinal preparations of Ayurveda in India and five traditional medicines in China, *Journal of Ethnopharmacology* 284: 114775.

Lilliu G. 1999, *Arte e religione della Sardegna prenuragica: idoletti, ceramiche, oggetti d'ornamento*, Sassari, Carlo Delfino Editore.

Longacre W.A., Xia J., Yang T. 2000, I Want to Buy a Black Pot, *Journal of Archaeological Method and Theory* 7: 273-293.

Lo Schiavo F. 1985, La preistoria, In Lo Schiavo F. (ed.), *Il Museo Sanna in Sassari*, Sassari, Banco di Sardegna: 19-62.

Lo Schiavo F. 1987, Grotta Verde 1979: un contributo sul Neolitico Antico della Sardegna, In *Atti della XXVI Riunione Scientifica dell'Istituto Italiano di Preistoria e Protostoria "Il Neolitico in Italia" (7-10 Novembre 1985)*, Firenze, Istituto Italiano di Preistoria e Protostoria: 845-858.

Lugliè C. 2004, La produzione lamellare in ossidiana nel Neolitico medio della Sardegna: un caso di studio da Bau Angius (Terralba, ORA), *Aristeo* 1: 33-46.

Lugliè C. 2005, *Le risorse litiche nel primo Neolitico della Sardegna centro-meridionale*, Cagliari, Edizioni AV.

Lugliè C. 2017, La comparsa dell'economia produttiva e il processo di neolitizzazione in Sardegna. In *Corpora delle antichità della Sardegna. La Sardegna preistorica. Storia, materiali, monumenti*, Firenze, Carlo Delfino Editore: 37-64.

Lugliè C. 2018a, *Your path led through the sea*…The emergence of Neolithic in Sardinia and Corsica, *Quaternary International* 470: 285-300.

Lugliè C. 2018b, Società di produzione e simbologia femminile: immagine del divino? In Fanni M., Sirigu M, Soro L., Congia C. (eds), Donna o Dea. Le raffigurazioni femminili nella preistoria e nella protostoria sarda, Cagliari, White Rocks Bay: 51-79.

Lugliè C. 2020a, Mobilità e affinità di gruppo, interazioni, reti di scambio, In Cossu T., Lugliè C. (eds.), *La preistoria in Sardegna. Il tempo delle comunità umane dal X al II millennio a.C.*, Nuoro, Ilisso: 50-53.

Lugliè C. 2020b, Le risorse minerarie regionali nel sistema tecnico neolitico, In Cossu T., Lugliè C. (eds.), *La preistoria in Sardegna. Il tempo delle comunità umane dal X al II millennio a.C.*, Nuoro, Ilisso: 68-72.

Lugliè C., Pinna V. 2012, Alla soglia del gesto: sequenze operative in incisioni su ciottolo del Neolitico Antico della Sardegna, In Lugliè C. (ed), *Atti della XLIV Riunione Scientifica La preistoria e la protostoria della Sardegna, Cagliari, Barumini, Sassari 23-28 novembre 2009*, Firenze, Istituto Italiano di Preistoria e Protostoria, vol. II: 477-483.

Lugliè C., Vacca G., Zara A. 2006. Il diaspro dell'Isola di San Pietro (Sardegna Sud-occidentale): prime osservazioni sullo sfruttamento e la circolazione durante il Neolitico In *Atti della XXXIX Riunione scientifica, Materie prime e scambi nella preistoria italiana, Firenze 25-27 novembre 2004*, Vol. II, Firenze, Istituto Italiano di Preistoria e Protostoria: 376-379.

Lugliè C., Le Bourdonnec F.-X., Poupeau G., Atzeni E., Dubernet S., Moretto P., Serani L. 2007, Early Neolithic obsidians in Sardinia (Western Mediterranean): the Su Carroppu case, *Journal of Archaeological Science* 34: 428-439.

Lugliè C., Paba R., Fanti L. 2019, Interazioni trans marine nel Neolitico medio della Sardegna. Componenti materiali e immateriali nell'orizzonte San Ciriaco a Su Forru de Is Sinzurreddus – Pau (Oristano), In Martorelli R. (ed.), *Know the sea to live the sea. Conoscere il mare per vivere il mare. Atti del convegno (Cagliari – Cittadella dei Musei, Aula Coroneo, 7-9 marzo 2019)*, Perugia, Morlacchi Editore U.P.: 235-249.

Maniatis Y., Tsirtsoni Z. 2004, Characterisation of a black residue in a decorated Neolithic pot from Dikili Tash, Greece: an unexpected result, *Archaeometry* 44: 229-239.

Marras V. 1999, Le culture prenuragiche nella collezione Vargiu di Villasor, *Studi Sardi* XXXI: 7-50.

Mastrotheodoros G. P., Beltsios K. G. 2022, Pigments – Iron-based red, yellow and brown ochres, *Archaeological and Anthropological Sciences* 14: 35.

Mazzieri P., Micheli R. 2014, Tradizioni funerarie e ornamenti personali. Alcune osservazioni dalla sfera VBQ emiliana alla luce delle ultime scoperte, *Rivista di Studi Liguri* LXXVII-LXXIX: 323-330.

Melosu B. 2020, L'uso dell'ocra rossa e di altri pigmenti colorati, In Cossu T., Lugliè C. (eds.), *La preistoria in Sardegna. Il tempo delle comunità umane dal X al II millennio a.C.*, Nuoro, Ilisso: 323.

Miller M. J., Whelton H. L., Swift J. A., Maline S., Hammann S., Cramp L. J.E., McCleary A., Taylor G., Vacca K., Becks F., Evershed R. P, Hastorf C. A. 2020, Interpreting ancient food practices: stable isotope and molecular analyses of visible and absorbed residues from a year-long cooking experiment, *Scientific Reports* 10: 13704.

Mioč U. B., Colomban Ph., Sagon G., Stojanović M, Rosić A. 2004, Ochre decor and cinnabar residues in Neolithic pottery from Vinča, Serbia, *Journal of Raman Spectrometry* 35: 843-846.

Odetti G. 2003, Riti sepolcrali dal Neolitico al Bronzo in Liguria, In *Atti della XXX Riunione Scientifica, Le comunità della Preistoria italiana. Studi e ricerche sul Neolitico e le età dei metalli*, Castello di Lipari, Chiesa di S. Caterina, 2 -7 giugno 2000, Volume II, Firenze, Istituto Italiano di Preistoria e Protostoria: 1065-1069.

Paba R., Thompson T.J.U., Fanti L., Lugliè C. 2021, Rising from the ashes: A multi-technique analytical approach to determine cremation. A case study from a Middle Neolithic burial in Sardinia (Italy), *Journal of Archaeological Science: Reports* 36: 102855.

Papakosta V., Smittenberg R. H., Gibbs K., Jordan P., Isaksson S. 2015, Extraction and derivatisation of absorbed lipid residues from very old and very small samples of ceramic potsherds for molecular analysis by gas-chromatography-mass spectrometry (GC-MS) and single compound stable carbon isotope analysis by gas-chromatography-combustion-.isotope ratio mass spectrometry (GC-C-IRMS), *Microchemical Journal* 123: 196-200.

Passeri L. 1970, Ritrovamenti preistorici nei Pozzi della Piana (Umbria), *Rivista di Scienze Preistoriche* 25: 225-251.

Pitzalis G., Fenu P., Martini F., Sarti L. 2004, Grotta Su Coloru: primi dati sui contesti culturali mesolitici e neolitici (scavi 1999-2003), *Sardinia Corsica Baleares Antiquae* I: 31-39.

Popelka-Filcoff R. S., Zipkin A. M. 2022, The archaeometry of ochre *sensu lato*: a review, *Journal of Archaeological Science* 137: 105530.

Pradeau J.-V. 2015, *Les matières colorantes au sein des systèmes techniques et symboliques au Néolithique (VIe et Ve millénaires BCE) dans l'arc liguro-provençal*. PhD thesis, Université Nice Sophia Antipolis, France.

Pradeau J.-V., Salomon H., Bon F., Mensan R., Lejay M., Regert M. 2014, Les matières colorantes sur le site aurignacien de plein air de Régismont-le-Haut (Poilhes, Hérault) : acquisition, transformations et utilisations, *Bulletin de la Société Préhistorique Française* 111: 631-658.

Pradeau J.-V., Binder D., Vérati C., Lardeaux J.-M., Dubernet S., Lefrais Y., Regert M. 2016, Procurement strategies of Neolithic colouring materials: Territoriality and networks from 6th to 5th millennia BCE in North-Western Mediterranean, *Journal of Archaeological Science* 71: 10-23.

Puglisi S. 1942, Villaggi sotto roccia e sepolcri megalitici della Gallura, *Bullettino di Paletnologia italiana* 5/6: 123-141.

Russell J., Sun M., Liang W., He M., Schroën Y., Zou W., Pommerening T., Wang M. 2021, An investigation of the pharmacological applications used for the Ancient Egyptian systemic model '*ra-ib*' compared with modern Traditional Chinese Medicine, *Journal of Ethnopharmacology* 265: 113115.

Quarta G., D’Elia M., Butalag K., Mariuccio L., Demortier G., Calcagnile L. 2006, An integrated accelerator mass spectrometry radiocarbon dating and ion beam analysis approach for the study of archaeological contexts, *Applied Physics A* 83: 605-609.

Quarta G., Aprile G., Ingravallo E., Tiberi I., Calcagnile L. 2018, Radiocarbon dates and XRF analyses from two prehistoric contexts in the Badisco area (Otranto-LE), *Measurements* 125: 279-283.

Rampazzi L., Cariati F., Tanda G., Colombini M. P. 2002, Characterisation of wall paintings in the Sos Furrighesos necropolis (Anela, Italy), *Journal of Cultural Heritage* 3: 237-240.

Rampazzi L., Campo L., Cariati F., Tanda G. 2007, Prehistoric wall paintings: the case of the Domus de Janas necropolis (Sardinia, Italy), *Archaeometry* 49: 559-569.

Reber E. A. 2021, Comparison of neutral compounds extraction from archaeological residues in pottery using two methodologies: a preliminary study, *Separations* 8: 6.

Reber E. A., Kerr M. T., Whelton H. L., Evershed R. P. 2019, Lipids from low-fired pottery, *Archaeometry* 61: 131-144.

Rifkin R. F. 2011, Assessing the efficacy of red ochre as an prehistoric hide tanning ingredient, *Journal of African Archaeology* 9: 131-158.

Roffet-Salque M., Dunne J., Altoft D. T., Casanova E., Cramp L. J.E., Smyth J., Whelton H. L., Evershed R. P. 2017, From the inside up: Upscaling the organic residue analyses of archaeological ceramics, *Journal of Archaeological Science: Reports* 16: 627-640.

Rueff B., Debels P., Vargiolu R., Zahouani H., Procopiou H. 2021, Reading ceramic surfaces: Characterisation of surface treatments towards functional identification of vases, *Journal of Archaeological Science: Reports* 38: 103021.

Salis G. 2020, Statuette antropomorfe e religiosità prenuragica. Spunti di riflessione alla luce dei rinvenimenti a Cannas di Sotto – Carbonia, In Melis M. G. (ed.), *Omaggio a Enrico Atzeni. Miscellanea di Paletnologia*, Quaderni del LaParS 4, Sassari, Università di Sassari: 239-253.

Salomon H. 2009, *Les matières colorantes au début du Paléolithique supérieur : sources, transformations et fonctions*, PhD thesis, Université Bordeaux I, France.

Salomon H., Chanteraud C., Chassin de Kergommeaux A., Monney J., Pradeau J.-V., Goemaere E., Coquinot Y., Chalmin E. 2021, A geological collection and methodology for tracing the provenence of Palaeolithic colouring materials, *Journal of Lithic Studies* 8: 1, DOI: https://doi.org/10.2218/jls.5540

Santoni V. 1982, Cabras – Cuccuru S’Arriu. Nota preliminare di scavo (1978, 1979, 1980), *Rivista di Studi Fenici* X: 103-110.

Santoni V. 2000, Alle origini dell'ipogeismo in Sardegna: Cabras-Cuccuru S'Arriu, la necropoli del Neolitico Medio, In *L'ipogeismo nel Mediterraneo: origini, sviluppo, quadri culturali. Atti del congresso internazionale, Sassari-Oristano 23-28 maggio 1994*, Muros, Stampacolor: 369-397.

Santoni V. 2012, Il Neolitico di Capo Sant’Elia – Cagliari, In Del Vais C. (ed.), *Epi Oinopa Ponton. Studi sul Mediterraneo Antico in ricordo di Giovanni Tore*, Oristano, S’Alvure: 97-120.

Santoni V. 2019, Il Neolitico medio recente della sacca 2 del 1989 di Cuccuru S’Arriu di Cabras-Oristano, in Melis M. G. (ed.), *Omaggio a Enrico Atzeni. Miscellanea di Paletnologia*, Sassari, Università di Sassari: 255-294.

Santoni V., Bacco G., Sabatini D. 1997, L'orizzonte Neolitico Superiore di Cuccuru s'Arriu di Cabras. Le sacche C.S.A. nn. 377, 380/1979 e N. 2/1989, In Campus L. (ed.), *La Cultura di Ozieri. La Sardegna e il Mediterraneo nel IV e III millennio a. C. Atti del II convegno di studi (Ozieri, 15-17 ottobre 1989)*, Ozieri, Il Torchietto: 277-295.

Serradimigni M. 2012, Le pintaderas nel quadro del Neolitico italiano: arte, simbolismo e funzionalità, *Preistoria Alpina* 46: 203-210.

Shemluck M. 1982, Medicinal and other uses of the *Compositae* by Indians in the United States and Canada, *Journal of Ethnopharmacology* 5: 303-358.

Schoepfer V.A., Burton E.D., 2021, Schwertmannite: A review of its occurrence, formation, structure, stability and interactions with oxyanions, *Earth-Science Reviews* 221: 103811. https://doi.org/10.1016/j.earscirev.2021.103811

Shoemaker A., Davies M., Moore H. 2017, Back to the Grindstone? The Archaeological Potential of Grinding-Stone Studies in Africa with Reference to Contemporary Grinding Practices in Marakwet, Northwest Kenya, *African Archaeological Review* 34: 1-21.

Siddall R. 2018, Mineral pigments in archaeology: their analysis and the range of available materials, *Minerals* 8: 201.

Skibo J.M. 2013, *Understanding Pottery Function*. New York, Springer.

Spades S., Russ J. 2005, GC-MS analysis of lipids in prehistoric rock paints and associated oxalate coatings from the lower Pecos Region, Texas, *Archaeometry* 47: 111-126.

Sparacello V., Panelli C., Rossi S., Dori I., Varalli A., Goude G. Starnini E., Biagi P. 2019, The re-discovery of Arma dell'Aquila (Finale Ligure, Italy): New insights on Neolithic funerary behavior from the sixth millennium BCE in the north-western Mediterranean, *Quaternary International* 512: 67-81.

Suryanarayan A., Méry S., Mazuy A., Regert M. 2022, Foodstuffs and organic products in ancient SE Arabia: preliminary results of ceramic lipid residue analysis of vessels from Hili 8 and Hili North Tomb A, al Ain, United Arab Emirates, Proceedings of the Seminar for Arabian Studies 51: 379-401.

Taçon P. SC. 2012, Ochre, clay, stone and art. The symbolic importance of minerals as life-force among aboriginal peoples of northern and central Australia, Inn Boivin N., Owoc M.A. (eds.), *Soils, stones and symbols: cultural perceptions of the mineral world*, London, UCL Press: 31-42.

Tanda G. 1980, Il Neolitico Antico e Medio della Grotta Verde, Alghero, in *Atti della XXII Riunione Scientifica dell'Istituto Italiano di Preistoria e Protostoria*, Firenze: 45-94,

Tanda G. 2003, L'uso del colore nella preistoria della Sardegna, *Atti della XXX Riunione Scientifica, Le comunità della Preistoria italiana. Studi e ricerche sul Neolitico e le età dei metalli*, Castello di Lipari, Chiesa di S. Caterina, 2 -7 giugno 2000, Volume I, Firenze, Istituto Italiano di Preistoria e Protostoria: 465-482.

Tanda G., Cariati F., Colombini M. P., Rampazzi L. 2003, Caratterizzazione delle pitture parietali presenti nella necropoli di Sos Furrighesos (Anela-SS), In *Studi in onore di Ercole Contu*, Sassari, Editrice Democratica Sarda: 61-71.

Teklay M., Thole J. T., Ndumbu N., Vries J., Mezger K. 2022, Mineralogical and chemical characterization of ochres used by the Himba and Nama people of Namibia, *Journal of Archaeological Science: Reports* 47: 103690.

Trump D. H. 1983, La grotta di Filiestru a Bonu Ighinu, Mara (SS), *Quaderni, Ministero per i beni culturali e ambientali, Soprintendenza ai Beni Archeologici per le Provincie di Sassari e Nuoro* 13, Sassari, Dessì.

Ucelli Gnesutta P. 2003, Testimonianze di culti funerari nella Grotta di Settecannelle (Ischia di Castro - Viterbo), In *Atti della XXX Riunione Scientifica, Le comunità della Preistoria italiana. Studi e ricerche sul Neolitico e le età dei metalli*, Castello di Lipari, Chiesa di S. Caterina, 2 -7 giugno 2000, Volume II, Firenze, Istituto Italiano di Preistoria e Protostoria: 1071-1075.

Ucchesu M., Sau S., Lugliè C. 2017, Crop and wild plant exploitation in Italy during the Neolithic period: New data from Su Mulinu Mannu, Middle Neolithic site of Sardinia, *Journal of Archaeological Science: Reports* 14: 1-11.

Van Gijn A., Verbaas A., Dekker J., Feisrami T., de Koning N., Spithoven M., Timmer T., Vernon F. 2020, Studying the life history of vessels. Creating a reference collection for microwear studies of potter, In Van Gijn A., Fries-

Knoblach, Stockhammer P. (eds), *Pots and practices. An experimental and microwear approach to Early Iron Age vessel biographies*, Leiden, Sidestone Press: 65-107.

Velliky E. C., Porr, M., Conard N. M. 2018, Ochre and pigment use at Hohle Fels cave: Results of the first systematic review of ochre and ochre-related artefacts from the Upper Palaeolithic in Germany, *PLOS One* 13: e0209874.

Vieugué J. 2014, Use-wear analysis of prehistoric pottery: methodological contribution from the study of the earliest ceramic vessels in Bulgaria (6100-5500 BC), *Journal of Archaeological Science* 41: 622-630.

Vieugué J., Salanova L., Regert M., Mirabaud S., Le Hô A.-S., Laval E. 2015, The consumption of bone powder in the Early Neolithic societies of Southeastern Europe: evidence of a diet stress? *Cambridge Archaeological Journal* 25: 495-511.

Volante N. 2015, Notizia preliminare sulla cava neolitica di cinabro sul Poggio di Spaccasasso nel Parco Regionale della Maremma (Alberese, GR), *Notiziario della Soprintendenza per i Beni Archeologici della Toscana*, 11: 429-440.

Wadley L. 2005, Putting ochre to the test: replication studies of adhesives that may have been used for hafting tools in the Middle Stone Age, *Journal of Human Evolution* 49: 587-601.

Whelton H. L., Hammann S., Cramp L. J. E., Dunne J., Roffet-Salque M., Evershed R. P. 2021, A call for caution in the analysis of lipids and other small biomolecules from archaeological contexts, *Journal of Archaeological Science* 132: 105397.

Zamagni B. 2007, Reperti in pietra non scheggiata, In Tozzi C., Weiss M. C. (eds), *Préhistoire et protohistoire de l'aire tyrrhénienne*, Ghezzano, Felici Editore: 127-133.

Zemour A. 2019, Trepanation and (ritual?) perimortem actions in the Neolithic period ad Grotta Patrizi (Lazio, Italy), *International Journal of Osteoarchaeology* 30: 80-89.

Zemour A., Binder D., Coppa A., Duday H. 2017, La sépulture au début du Néolithique (VI$^{e}$ millénaire et première moitié du V$^{e}$ millénaire cal BC) en France méridionale et en Italie : de l'idée d'une « simple » fosse à une typologie architecturale, *Bulletins et Mémoires de la Société d'Anthropologie de Paris* 29: 94-111.